PB-231 257

COLOR REMOVAL FROM KRAFT MILL EFFLUENTS BY ULTRAFILTRATION

H. A. Fremont, et al

Champion International Corporation
Hamilton, Ohio

December 1973

DISTRIBUTED BY:

NTIS
National Technical Information Service
U. S. DEPARTMENT OF COMMERCE
5285 Port Royal Road, Springfield Va. 22151

PB 231 257

1 Accession Number
W74-06521

2 Subject Field & Group
05D, 03E

SELECTED WATER RESOURCES ABSTRACTS
INPUT TRANSACTION FORM

5 Organization
Champion International Corporation,
~~Knightsbridge~~
Hamilton, Ohio. ~~45020~~

6 Title
COLOR REMOVAL FROM KRAFT MILL EFFLUENTS BY ULTRAFILTRATION,

10 Author(s)
H. A. Fremont
D. C. Tate
R. L. Goldsmith ~~(Abcor, Inc.~~
~~Cambridge, Mass.)~~

16 Project Designation
EPA Project S 800 261

21 Note
Copy Available from GPO Sup Doc
as EP1.23:660/2-73-019, $ 2.40;
microfiche from NTIS as PB
$1.45

22 Citation
Environmental Protection Agency, Technology Series report ~~number,~~ EPA-660/2-73-019, December 1973. 240p, 58 fig, 35 tab.

23 Descriptors (Starred First) *Pulp and Paper Industry, ~~*Pulp Mill~~ Effluents, *Waste Water Treatment, *Pilot Plants, *Cost Analyses, *Pulp Wastes, ~~*Ultrafiltration,~~ *Filtration, *Membrane Processes, *Organic ~~Removal~~ wastes, *Reverse Osmosis, *Color, ~~Removal,~~ Operation and Maintenance, Incineration, *Water Reuse, *North Carolina, Recycling

25 Identifiers (Starred First) *Ultrafiltration Process, *Waste Water Recycle, *Color Removal, *Organic Removal, *Bleach Caustic Extraction Effluent, *Kraft Decker Effluent, Canton (N. C.)

27 Abstract The purpose ~~of this program~~ was to examine ultrafiltration as a means of reducing color in kraft mill effluents more efficiently and/or more economically than the presently available method. ~~The scope of~~ the program included the six month operation of a 10,000 gpd pilot plant at the Champion Papers' Canton, North Carolina, pulp and paper mill.

The major experimental effort dealt with treatment of pine bleaching caustic extraction filtrate with lesser emphasis on unbleached pine and hardwood pulp washing Decker effluents. Four experimental aspects of the process were evaluated: feed pretreatment, ultrafiltration, concentrate disposal and water reuse potential.

The process color removal efficiency was satisfactory. For all influent studied typical results were 90% color removal with 98.5-99% water recovery. The total operating costs, including amortization and exclusive of credits for a one million gallon per day treatment plant are estimated to be $0.58/ton bleached pulp for pine caustic extraction filtrate influent and $0.33/ton total pulp for Decker effluents as influent. (EPA)

Abstractor H. A. Fremont
Institution Champion International Corporation

WR 102 (REV. JULY 1969)
WRSIC

SEND TO: WATER RESOURCES SCIENTIFIC INFORMATION CENTER
U.S. DEPARTMENT OF THE INTERIOR
WASHINGTON, D. C. 20240

U.S. GOVERNMENT PRINTING OFFICE: 1974—546-314:199

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
U S Department of Commerce
Springfield VA 22151

EPA-660/2-73-019
December 1973

COLOR REMOVAL FROM KRAFT MILL EFFLUENTS BY ULTRAFILTRATION

by

H. A. Fremont
D. C. Tate
R. L. Goldsmith

Project S800261
Program Element 1B2037

Project Officer

Mr. Edmond P. Lomasney
Environmental Protection Agency
Atlanta, Georgia 30309

prepared for the

OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 - Price $2.10

RESEARCH REPORTING SERIES

Research reports of the Office of Research and Monitoring, Environmental Protection Agency, have been grouped into five series. These five broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The five series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies

This report has been assigned to the ENVIRONMENTAL PROTECTION TECHNOLOGY series. This series describes research performed to develop and demonstrate instrumentation, equipment and methodology to repair or prevent environmental degradation from point and non-point sources of pollution. This work provides the new or improved technology required for the control and treatment of pollution sources to meet environmental quality standards.

EPA Review Notice

This report has been reviewed by the Environmental Protection Agency and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Environmental Protection Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

ABSTRACT

Reduction of color in pulp mill effluents by ultrafiltration has been examined with a 10,000 gallon per day (gpd) pilot plant. Treated streams included Decker effluents and pine bleachery caustic extraction filtrate, which together comprise about 80% of the color from a bleached kraft mill.

High color removal (90-97%) was demonstrated when operating at water recovery ratios of 98.5-99%. Pilot plant capacity (membrane flux) was 15-20 gal./day-ft^2 when operation proceeded smoothly. However, plugging of the membrane cartridges by residual particulates (even after precoat filtration) was troublesome.

Several prefiltration, concentrate disposal, and water reuse alternatives were evaluated.

Full-scale plant designs, and approximate capital and operating costs were estimated for systems of 1 and 2 MM gpd capacity. Capital costs are about $700,000 for a 1 MM gpd plant, and $1,200,000 for a 2 MM gpd plant. Corresponding operating costs are about 45¢/Mgal. (1 MM gpd) and 38¢/Mgal. (2 MM gpd).

Additional pilot plant tests are recommended to demonstrate long-term solutions to the particulate problem and long-term membrane cartridge life.

This report was submitted in fulfillment of Project Number S800261, by Champion International Corp. under the partial sponsorship of the Environmental Protection Agency. Work was completed as of May, 1973.

TABLE OF CONTENTS

Page

I. SUMMARY AND CONCLUSIONS 1

A. NATURE OF PROBLEM 1

B. NATURE OF STREAMS TREATED 1

C. PROCESS CONCEPT 2

D. PROGRAM RESULTS 3

E. FULL-SCALE PLANT DESIGN AND PROCESS COSTS 5

II. RECOMMENDATIONS 7

IIII. BACKGROUND 9

A. NATURE OF PROBLEM AND PROJECT GOALS 9

B. PROCESS DESCRIPTION 12

IV. PILOT PLANT DESCRIPTION 25

A. GENERAL 25

B. PRETREATMENT SEQUENCES 28

C. OTHER SYSTEM MODIFICATIONS 30

D. DESCRIPTION OF MEMBRANE CARTRIDGES 33

E. SAMPLING AND ANALYTICAL PROCEDURES 50

V. RESULTS AND DISCUSSION 53

A. FEED PRETREATMENT AND CHARACTERISTICS 53

B. REJECTION DATA AND EVALUATION 80

C. ULTRAFILTRATION RATE DATA AND EVALUATION 88

D. PRESSURE DROP DATA 115

TABLE OF CONTENTS
(continued)

Page

E. IMPORTANT FACTORS CONTROLLING MEMBRANE FLUX 121

F. CLEANING PROCEDURES AND EFFICIENCY 125

G. MODULE MECHANICAL FAILURES 129

H. INCINERATOR STUDIES 134

VI. FULL SCALE PLANT DESIGN AND COSTS 139

A. FIRST STAGE PINE CAUSTIC EXTRACTION FILTRATE 139

B. DECKER EFFLUENTS 173

VII. WATER REUSE 189

A. PINE CAUSTIC EXTRACTION FILTRATE PERMEATE 189

B. DECKER EFFLUENT PERMEATES 189

VIII. ACKNOWLEDGEMENTS 191

IX. REFERENCES 193

APPENDIX A - DETAILED PILOT PLANT PROCESS DESCRIPTION 197

APPENDIX B - ULTRAFILTRATION PILOT PLANT SPIRAL MEMBRANE CARTRIDGE IDENTIFICATION 217

APPENDIX C - DETAILS OF FILTERS USED IN PILOT PLANT PROGRAM 223

APPENDIX D - MECHANICAL PROBLEMS OF DIFFERENT SPIRAL MEMBRANE CARTRIDGES 231

APPENDIX E - SLIME ANALYSIS 237

FIGURES

		Page
1.	SIMPLIFIED ULTRAFILTRATION FLOW SCHEMATIC	13
2.	CAUSTIC EXTRACTION FILTRATE--FLOW SCHEMATIC	18
3.	TYPICAL MATERIAL BALANCE FOR TREATMENT OF CAUSTIC EXTRACTION FILTRATE BY ULTRAFILTRATION	19
4.	DECKER EFFLUENTS--FLOW SCHEMATIC	21
5.	TYPICAL MATERIAL BALANCE FOR TREATMENT OF HARDWOOD DECKER EFFLUENT BY ULTRAFILTRATION	22
6.	SIMPLIFIED PILOT PLANT FLOW SCHEMATIC	26
7.	FEED PRETREATMENT FLOW SCHEMATIC	29
8.	BAUER HYDRASIEVE	34
9.	TOP OF 500 GAL. FEED TANK	35
10.	ACID PUMP AND ACID DRUM	36
11.	DETERGENT MIXING TANK AND PRECOAT MIXING TANK WITH MIXER	37
12.	PRETREATMENT SEQUENCE	38
13.	DETAILS OF SPARKLER FILTER	39
14.	SHRIVER FILTER PRESS	40
15.	100 GAL. FILTRATE SURGE TANK	41
16.	SINGLE CARTRIDGE AND FILTERS	42
17.	SPARKLER VELMAC DISC FILTER	43
18.	ULTRAFILTRATION UNIT	44
19.	ULTRAFILTRATION UNIT (TOP VIEW)	45
20.	DETAILS OF CONTROL PANEL RIGHT SIDE	46
21.	DETAILS OF CONTROL PANEL LEFT SIDE	47

FIGURES

(continued)

Page

22. SPIRAL WOUND MODULE DESIGN 48

23. SULFURIC ACID REQUIREMENT TO NEUTRALIZE CAUSTIC EXTRACTION FILTRATE 54

24. SULFURIC ACID REQUIREMENT TO NEUTRALIZE PINE DECKER EFFLUENT 55

25. SULFURIC ACID REQUIREMENT TO NEUTRALIZE HARDWOOD DECKER EFFLUENT 56

26. EFFECT OF pH ON COLOR OF PINE CAUSTIC EXTRACTION FILTRATE 58

27. SUSPENDED SOLIDS OF CAUSTIC EXTRACTION FILTRATE 70

28. SUSPENDED SOLIDS OF PINE DECKER EFFLUENT 71

29. SUSPENDED SOLIDS OF HARDWOOD DECKER EFFLUENT 72

30. TOTAL SOLIDS OF PINE CAUSTIC EXTRACTION FILTRATE AND PINE DECKER EFFLUENTS 73

31. TOTAL SOLIDS OF HARDWOOD DECKER EFFLUENT 74

32. COLOR OF PINE CAUSTIC EXTRACTION FILTRATE AND PINE DECKER EFFLUENT 75

33. COLOR OF HARDWOOD DECKER EFFLUENT 76

34. EFFECT OF FEED PRETREATMENT ON COLOR OF PINE CAUSTIC EXTRACTION FILTRATE 78

35. REJECTION AND CONVERSION DATA 81

36. MEMBRANE FLUX FOR STAGE 1a 94

37. MEMBRANE FLUX FOR STAGE 1b 95

38. MEMBRANE FLUX FOR STAGE 1c 96

39. MEMBRANE FLUX FOR STAGE 2 97

FIGURES
(continued)

		Page
40.	MEMBRANE FLUX FOR STAGE 3	98
41.	MEMBRANE FLUX FOR STAGE 4	99
42.	MEMBRANE FLUX FOR STAGE 5	100
43.	EFFECT OF PREFILTRATION EFFICIENCY ON ULTRAFILTRATION RATE	102
44.	ULTRAFILTRATION RATE DATA: STAGE 1b	108
45.	ULTRAFILTRATION RATE DATA: STAGE 2	110
46.	FLUX VS. TIME FOR STAGES 1a AND 1b	114
47.	STAGE 1 PRESSURE DROP	116
48.	STAGE 2 PRESSURE DROP	117
49.	STAGE 3 PRESSURE DROP	118
50.	STAGE 4 PRESSURE DROP	119
51.	STAGE 5 PRESSURE DROP	120
52.	CLEANING EFFICIENCY OF STAGE 1b MEMBRANES	130
53.	CLEANING EFFICIENCY OF STAGE 3 MEMBRANES	131
54.	STAGE 3 COMPACTION CURVE	132
55.	SIMPLIFIED FLOW SCHEMATIC: TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE	140
56.	FLOW SCHEMATIC FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE: LOW FLOW CASE	141
57.	FLOW SCHEMATIC FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE: HIGH FLOW CASE	142
58.	SIMPLIFIED FLOW SCHEMATIC: TREATMENT OF DEKCER EFFLUENTS	174

TABLES

Page

1. WASTE CHARACTERISTICS AT THE NORTH CAROLINA MILL 11
2. FILTERS USED 31
3. CHARACTERISTICS OF MEMBRANE CARTRIDGES 49
4. ANALYTICAL PROCEDURES 51
5. PARTICLE SIZE DISTRIBUTION OF SUSPENDED SOLIDS IN PINE CAUSTIC EXTRACTION FILTRATE 61
6. PERFORMANCE - MAIN FILTERS 63
7. TESTS WITH DIFFERENT FILTER AIDS ON SPARKLER 15.3 SQ FT LEAF FILTERS 67
8. SUMMARY OF FEED CHARACTERISTICS 69
9. WASTE CHARACTERISTICS AT THE NORTH CAROLINA MILL - DETAILED ANALYSES 79
10. REJECTION 82
11. COMPOSITION DATA: PINE DECKER EFFLUENT SAMPLES 85
12. COMPOSITION DATA: HARDWOOD DECKER EFFLUENT SAMPLES 86
13. COMPOSITION DATA: PINE CAUSTIC EXTRACTION FILTRATE SAMPLES 87
14. MEMBRANE FLUX BY STAGE DURING PILOT PLANT PROGRAM 89
15. EFFECT OF FEED PREFILTRATION ON CLEANING 127
16. CASES FOR CAPITAL COST ESTIMATES, PINE CAUSTIC EXTRACTION FILTRATE 145
7. CHARACTERISTICS OF MEMBRANE CARTRIDGES 147

TABLES
(continued)

Page

18. CHARACTERISTICS AND COSTS OF MEMBRANE MODULES 148

19. ULTRAFILTRATION SECTION DESIGN DETAILS AND COSTS--CASE 1 153

20. ULTRAFILTRATION SECTION DESIGN DETAILS AND COSTS--CASE 2 158

21. SUMMARY OF INSTALLED CAPITAL COST ESTIMATES PLANT TO TREAT PINE CAUSTIC EXTRACTION FILTRATE 162

22. OPERATING COSTS FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE, CASE 1 163

23. OPERATING COSTS FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE, CASE 2 164

24. OPERATING COSTS FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE, CASE 3 165

25. OPERATING COSTS FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE, CASE 4 166

26. OPERATING COSTS FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE, CASE 5 167

27. DAILY INCREMENTAL OPERATING COSTS, TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE 168

28. CASES FOR CAPITAL COST ESTIMATES, DECKER EFFLUENTS 175

29. INSTALLED CAPITAL ESTIMATES--PLANT TO TREAT DECKER EFFLUENTS 176

30. OPERATING COSTS FOR TREATMENT OF DECKER EFFLUENT, CASE 6 178

31. OPERATING COSTS FOR TREATMENT OF DECKER EFFLUENT, CASE 7 179

TABLES

(continued)

Page

32. OPERATING COSTS FOR TREATMENT OF DECKER EFFLUENT, CASE 8 180

33. INCREMENTAL OPERATING COSTS FOR TREATMENT OF DECKER EFFLUENT, CASE 9 181

34. INCREMENTAL OPERATING COSTS FOR TREATMENT OF DECKER EFFLUENT, CASE 10 182

35. DAILY INCREMENTAL OPERATING COSTS, TREATMENT AND REUSE OF DECKER EFFLUENTS 183

SECTION I

SUMMARY AND CONCLUSIONS

A. NATURE OF PROBLEM

At present, in kraft paper mills color pollutants are not substantially removed by conventional biological waste treatment methods. The available means, such as lime precipitation and carbon or resin adsorption are highly expensive.

In the Champion Papers' North Carolina mill, approximately 60% of the mill color discharge is contained in the bleach plant caustic extraction filtrate, and 20% in the Decker effluents. These numbers are fairly typical for a bleached kraft mill. Thus, reduction of color from these two streams can greatly reduce the overall color discharge from a kraft mill.

The purpose of this program has been to examine ultrafiltration as a means of reducing color in kraft mill effluents more efficiently and/or more economically than the presently available methods. The scope of the program included the six month operation of a 10,000 gpd pilot plant at the Champion Papers' North Carolina mill.

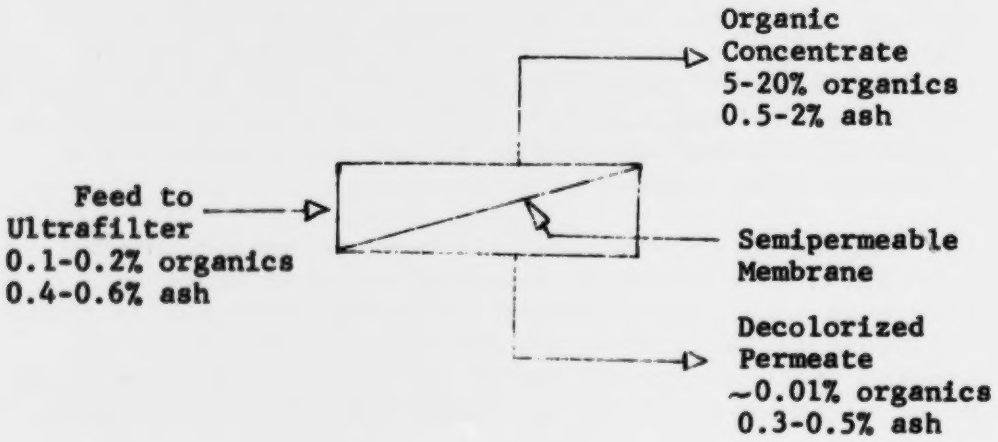
B. NATURE OF STREAMS TREATED

The major experimental effort dealt with treatment of pine caustic extraction filtrate, with lesser emphasis placed on pine and hardwood Decker effluents. All three streams are highly alkaline (pH 10-12) and hot ($120^{o}F$-$135^{o}F$), and require neutralization (to pH7) and cooling (to $100^{o}F$-$105^{o}F$) before treatment by ultrafiltration. These limits are imposed by the characteristics of current cellulose acetate membranes, which will not exhibit long (economical) life if exposed to high pH and high temperature.

These streams also contain substantial quantities of particulates (e.g. 100-300 ppm), of which 50% are smaller than 10μ. To obtain acceptable membrane equipment performance it is essential to employ membrane equipment (configuration) which is not susceptible to plugging by particulates. In addition, flocculation occurs in a freshly filtered feed on "aging".

C. PROCESS CONCEPT

Ultrafiltration, can selectively concentrate color bodies and organics contained in the streams. A simplified flow schematic is shown below:


Organic
Concentrate
5-20% organics
0.5-2% ash
Feed to
Ultrafilter
0.1-0.2% organics
0.4-0.6% ash
Semipermeable
Membrane
Decolorized
Permeate
~0.01% organics
0.3-0.5% ash

Desirable features of the process (compared to reverse osmosis) are:

- Since only 5-30% of the dissolved solids are returned in the concentrate, very high water recovery (e.g. 99%) can be achieved;
- A low-volume, high-organic-solids concentrate is obtained, which has substantial heating value if ultimate disposal is by burning;
- Low-pressure systems (e.g. 100 psig) can be employed; and
- High capacity can be achieved since higher-flux membranes can be used.

Limitations of the process are:

- The effluent is not demineralized, and the residual salt content (especially chlorine in caustic extraction filtrate) limits reuse potential; and
- Color removal is typically 90-97%, somewhat lower than can be achieved by reverse osmosis.

For all three streams examined, pretreatment (neutralization, temperature reduction, and filtration) is required.

D. PROGRAM RESULTS

A pilot plant was operated from August, 1972 through February, 1973. Operation was normally 24 hrs/day, 7 days/week. During this period four experimental aspects of the process were evaluated:

- Feed pretreatment (especially filtration);
- Ultrafiltration (separation efficiency and capacity);
- Concentrate disposal (incineration); and
- Water reuse potential.

Feed pretreatment work focused primarily on means to remove particulates, by surface, packed-bed, and precoat filtration, to obtain satisfactory ultrafiltration performance. Using filtration which removed particles of about 2μ and larger, 50-80% particulate removal was achieved.

The ultrafiltration system membranes were in a spiral wound configuration, and were obtained from three vendors (T. J. Engineering, Gulf Environmental Systems, and Eastman Chemical Products). Most membrane cartridges had the standard "mesh" flow-channel spacer; a few had "corrugated" spacers. Operation was usually at about 100°F, 100 psig, and pH 6-7.

Color removal efficiency was satisfactory; typical results are:

Influent	% Water Recovery	% Solids In Concentrate	% Color Removal
Pine caustic extraction filtrate	98.5-99	15-20	90-92
Pine Decker effluent	98.5-99	5-8	95-97
Hardwood Decker effluent	98.5-99	5-8	95-97

Capacity (membrane flux) was variable. When operation proceeded smoothly, fluxes of 15-20 gallons/day-ft^2 of membrane were achieved. At other times, fluxes were substantially lower. Important factors which reduced flux were:

- Reversible membrane surface fouling by colloidal and macromolecular feed constituents. This was reversed by water flushing and detergent cleaning;
- Irreversible membrane "compaction". This was minor; and
- Reversible and irreversible particulate collection within the membrane cartridges.

The latter limitation is the most serious problem encountered in the test program. Due to incomplete particulate removal in the pretreatment operation, particulates collected in the membrane cartridges. This was especially severe for cartridges with "mesh" spacers, but not for the "hydrodynamically-clean" corrugated spacers. Manifestations of cartridge plugging were:

- Occlusion and inactivation of membrane surface, with reduced capacity;
- High pressure drop across the cartridges which resulted in: (a) cartridge deformation and (b) seal failure;
- Flow maldistribution within cartridges and between cartridges in parallel, which aggravated cartridge plugging by particulates; and
- Various modes of module mechanical failures.

Particulate plugging can be minimized substantially by:

- Operation at high flow, which prevents particulate collection;
- Regular and efficient cartridge cleaning; and
- Use of cartridge flow-channel spacers which are not susceptible to particulate collection (e.g. corrugated spacers).

Concentrate disposal for pine caustic extraction filtrate by incineration or evaporation and admixture with primary sludge was demonstrated. Return of the concentrates from Decker effluents to the weak black liquor system was chosen as an optimum means of disposal.

Water reuse potential was examined. Analyses of treated effluents and comparison to mill water standards led to the following conclusions; treated effluent from pine caustic extraction filtrate has limited or no reuse potential due to a high chloride content and some residual color; treated water from the Decker effluents may be beneficially reused in pulp washing.

E. FULL-SCALE PLANT DESIGN AND PROCESS COSTS

A series of design cases were examined. Design parameters included:

- Plant capacity (1 MM gpd and 2 MM gpd);
- Membrane type (mesh and corrugated spacers); and
- Different prefiltration alternatives.

The key cost factor was plant capacity, as expected, since treatment costs are nearly proportional to the volume treated and not the contaminant loading.

The installed capital costs are estimated to be:

Flow	Pine Caustic Extraction Filtrate	Decker Effluents
1 MM gpd	$770,000	$690,000
2 MM gpd	$1,250,000	$1,100,000

The difference is due to concentrate disposal equipment required for the pine caustic extraction filtrate.

The total operating costs (including amortization) are estimated to be:

Flow	Pine Caustic Extraction Filtrate	Decker Effluents
1 MM gpd	46¢/M gal. = $0.58/ton bleached pulp	44¢/M gal. = $0.33/ton total pulp
2 MM gpd	35¢/M gal. = $0.88/ton bleached pulp	37¢/M gal. = $0.55/ton total pulp

SECTION II

RECOMMENDATIONS

The results of the project studies using the nominal 10,000 gpd ultrafiltration pilot plant demonstrate that the processing system is technically sound as a means for reducing color from the pine caustic extraction filtrate of a pulp bleachery and from the Decker effluents from pulp washing. Mechanical difficulties, however, were encountered which should be resolved. These problems and unknown variables are highly critical in developing reliable cost estimates for full scale installations.

It is recommended that pilot plant studies be continued for the purpose of confirming the present results over a longer time span, and further examining the cost sensitive areas of the system. More specifically, these additional studies could accomplish the following tasks:

1. Examine membrane cartridges from several manufacturers for long-term reliability.
2. Verify optimum operating conditions such as feed flow rates, pH, temperatures and operating pressures.
3. Further specify prefiltration conditions for each influent.
4. Test any new membranes that may become commercially available and which could afford the elimination of the neutralization and cooling treatments of the influent.
5. Obtain more extensive operating experience using Decker effluents.
6. The information from items 1 through 5 above could result in redesigning a full scale plant and assessing more reliably capital and operating cost estimates for such a plant.

SECTION III

BACKGROUND

A. NATURE OF PROBLEM AND PROJECT GOALS

The 121 kraft pulp mills in the United States produce about 85% of the chemical wood pulp consumed. In these pulping operations a substantial volume of wastewater is discharged, typically about 25,000 gallons per ton of pulp. Of concern are the pH, temperature, BOD, and color loading of this effluent. Conventional and generally inexpensive techniques are adequate for waste treatment except for color removal. It may also be noted that conventional waste treatment does not provide for water reuse or chemical recovery and reuse, and, as such, is not conducive to eventual close-loop operations.

Color bodies found in pulp mill wastes unfotunately are resistant to biological degradation. The effluent color is due primarily to lignin and its degraded products which are chemically stable and are intractible to separation by presently proven commercial processes. Consequently, new treatment techniques for color removal are undergoing active development and actual plant scale demonstration. Promising processes developed include chemical precipitation (1-10), including lime precipitation (stoichiometric and massive lime), adsorption (11-14), and reverse osmosis and ultrafiltration (15-25). For controlling color from bleaching effluents, it is also possible to modify the bleaching sequence to CHE .... from CEH ...; where C= chlorination, H =hypochlorite bleaching, and E = caustic extraction.

Segregation of mill wastes is often practiced and it is likely that segregation of waste streams by color will eventually be required for adequate waste treatment. Tertiary treatment systems which could not be considered cost-wise for treatment of the total effluent, might be applicable if the bulk of the color is contained in a relatively small fraction of the mill effluent.

For example, in Champion Papers' North Carolina mill, about 60% of the mill color is present in 2 x 10^6 gpd

Preceding page blank

of bleachery first-stage pine caustic extraction filtrate. This flow amounts to only about 4% of the total wastewater. Thus, if color could be removed from this stream at total operating costs of $450 to $800 per day, over 60% of the mill color could be removed at a cost of about 0.9¢ to 1.6¢/1000 gal. of total effluent or about 55¢ to $1.00 per ton of bleached pine pulp.

The second most important controllable source of color in a kraft mill is the decker effluent. This waste is present in all kraft mills, while the pine caustic extraction filtrate is found only in mills producing bleached pulp. At the North Carolina mill, approximately 2×10^6 gpd of mixed pine and hardwood decker effluents are currently discharged. This waste contributes about 20% of the mill color. Thus, decker effluents are another case of interest for segregated waste treatment.

Ranges of flow and composition for these streams at the North Carolina mill are given in Table 1.

The program described in this report encompasses the initial phase of a two-phase project for the development and demonstration of ultrafiltration as a means of color removal from kraft mill effluents. The site of the project is Champion Papers' North Carolina mill which is a representative bleached kraft mill. Phase I of the project (the subject of this report) included onsite operation of a nominal 10,000 gpd membrane demonstration plant to supplement data from previous pilot tests and to specify more accurately design bases for a full-scale demonstration plant. Additional studies determined requirements for feed pretreatment prior to ultrafiltration, disposal of the concentrated wastes produced by ultrafiltration, and the potential for water reuse.

Briefly the project objectives have been threefold.

1. To demonstrate with commercially-available equipment the effectiveness of ultrafiltration to reduce color in first-stage pine caustic extraction filtrate and decker effluents to low levels;

TABLE 1

WASTE CHARACTERISTICS AT THE NORTH CAROLINA MILL

Waste	Flowrate*, Million Gal/Day	pH	Color, ppm	Total Solids, ppm	Suspended Solids, ppm
Pine Caustic Extraction Filtrate	1.3 to 2.1	11.5 to 11.8	12,000 to 50,000; Avg. ∿ 28,000	4,000 to 14,000; Avg. ∿ 7,000	50 to 500; Avg. ∿ 80
Pine Decker Effluent	0.5 to 2.7; Avg. = 0.5	10.7 to 11.0	5,000 to 10,000; Avg. ∿ 6,000	1,500 to 6,000; Avg. ∿ 2,400	50 to 250; Avg. ∿ 80
Hardwood Decker Effluent	1.5 to 4.1; Avg. = 1.5	10.0 to 10.6	4,000 to 24,000; Avg. ∿ 11,000	1,200 to 5,000; Avg. ∿ 3,000	100 to 500; Avg. ∿ 200

* maximum flow is future projection

2. To examine the potential for reuse of purified effluents and means of disposal of the concentrated wastes produced by the membrane process; and

3. To demonstrate that process economics will be sufficiently attractive to lead to widespread adoption of this pollution abatement process in the industry.

B. PROCESS DESCRIPTION

1. Principles of Ultrafiltration

Ultrafiltration is a membrane process for concentration of dissolved materials in aqueous solution. A semipermeable membrane is used as the separating agent and pressure as the driving force. In an ultrafiltration process (Figure 1), a feed solution is fed into the membrane unit, where water and certain solutes pass through the membrane under an applied hydrostatic pressure. The solutes whose sizes are larger than the pore size of the membrane are retained and concentrated. The pore structure of the membrane thus acts as a molecular filter, passing some of the smaller solutes and retaining the larger solutes. The pore structure of this molecular filter is such that it does not become plugged because the solutes are rejected at the surface and do not penetrate the membrane. Furthermore, there is no continuous buildup of a filter cake which has to be removed periodically to restore flux through the membrane since concentrated solutes are removed in solution. Many ultrafiltration applications involve the retention of relatively high molecular weight solutes accompanied by the removal through the membrane of lower molecular weight impurities. Thus concentration of specific solution components can be achieved.

Considerations important for determining the technical and economic feasibility of ultrafiltration as applied to a specific process are the rate of solution transport through the membrane (flux) and the separation efficiency (rejection). Factors which control flux and rejection have been described elsewhere (26,27).

Membrane processes for treating pulp mill wastes have been under development for several years, focusing primarily on reverse osmosis. In reverse osmosis all

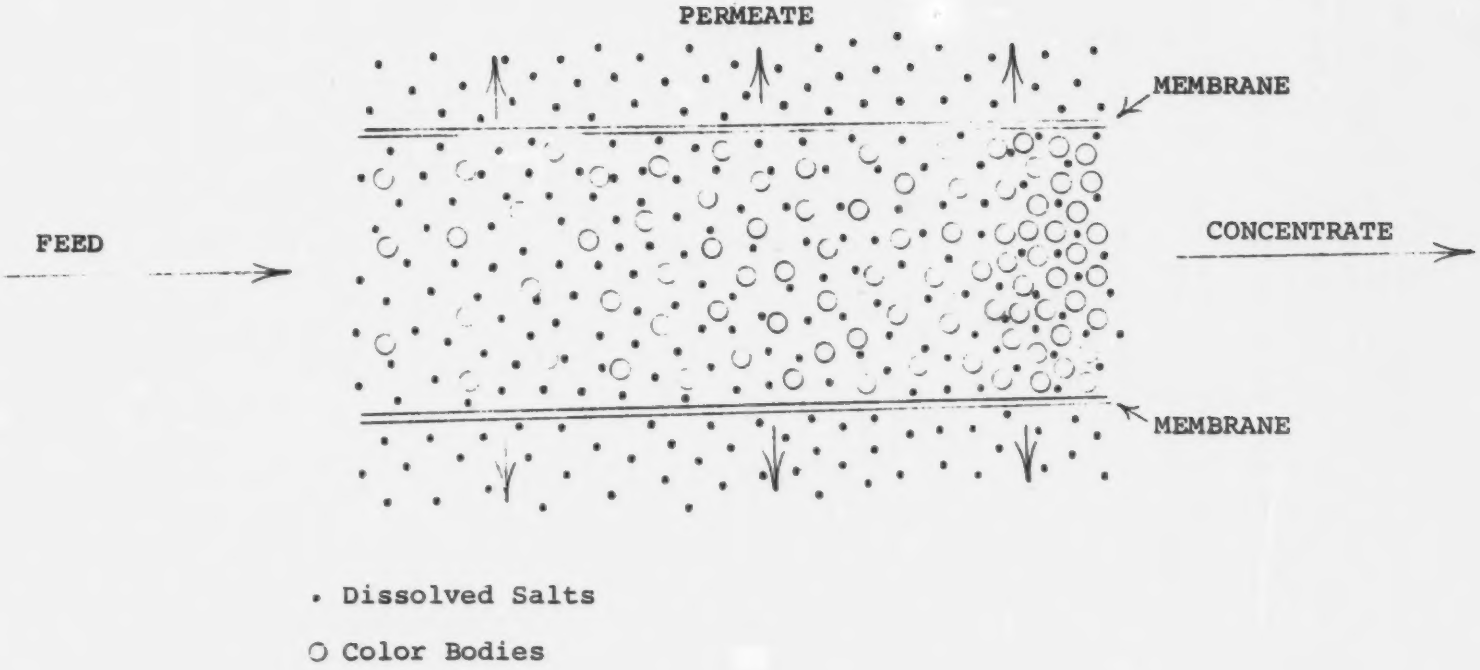

PERMEATE
MEMBRANE
FEED
CONCENTRATE
MEMBRANE
• Dissolved Salts
○ Color Bodies

FIGURE 1: SIMPLIFIED ULTRAFILTRATION FLOW SCHEMATIC

dissolved solutes are concentrated, and a demineralized aqueous effluent is produced. Ultrafiltration is, in fact, a variation of reverse osmosis. The fundamental difference relates to the retention properties of the membranes: ultrafiltration membranes do not retain salts and other low molecular weight solutes.

There are several potential advantages of ultrafiltration when compared to reverse osmosis, which are:

a. The use of ultrafiltration membranes leads to operation at lower pressures, typically 50 to 300 psig. The strength requirements of the membrane system are much less stringent than those for reverse osmosis sytems, which typically operate above 400 psig. At the lower pressures used in ultrafiltration, power costs are less; membranes can last longer since the rate of "compaction" may be reduced; and lower capital costs can be achieved due to less demanding requirements for pressure vessels, pumps, etc. Reliability is also an important consideration, and system failure observed in reverse osmosis tests to date can be tied to the high operating pressures used. Important factors have been membrane compaction (resulting in reduced capacity), membrane catastrophic failure (membrane support rupture), and pump failure.

b. With reverse osmosis the feed pine caustic extraction filtrate or decker effluent can be concentrated only to 8 to 10% total solids, or about 20-fold. This is due to limitations of both membrane fouling and a buildup in feed solution osmotic pressure. Both problems are of substantially less importance for ultrafiltration, in which very high volumetric concentration ratios are obtainable, up to or exceeding 200-fold. This is due primarily to a relatively slow increase in feed solids content with concentration by ultrafiltration. That is, only a small portion of the feed solids, specifically the higher molecular-weight organics, is retained by the ultrafiltration membrane. Note that about 80% of the dissolved solids in the wastes of interest are low molecular-weight salts. Since the retained solutes have relatively high molecular weights, osmotic pressure limitations are of minor importance, and solids levels up to 20% can be achieved in low-pressure ultrafiltration.

c. Disposal cost of an ultrafiltration concentrate by incineration or other means would be substantially less than that for a reverse osmosis concentrate, since a substantially smaller volume must be treated. For example, in treating 2×10^6 gpd, about 100,000 gpd of reverse osmosis concentrate would be generated, but only 10,000 gpd of ultrafiltration concentrate. Furthermore the high organic content of the latter provides substantial heating value, almost sufficient in itself to sustain combustion. A major fuel cost would be required to burn a reverse osmosis concentrate which contains primarily inorganics. In addition a high chlorine content in a reverse osmosis concentrate of pine caustic extraction filtrate could cause severe corrosion problems during incineration, and would require extensive off-gas scrubbing to remove volatile chloride particulates.

d. Membrane life data in the field in other applications has shown that ultrafiltration membranes are less susceptible than reverse osmosis membranes to deterioration in flux and rejection due to alkaline hydrolysis and/or compaction under pressure.

The major disadvantage of ultrafiltration vis-a-vis reverse osmosis lies in the quality of the aqueous effluent produced by the membrane system. Two factors are of importance. First, since ultrafiltration does not demineralize that waste treated, the effluent does contain residual salts. As discussed below, this should not present any problem in the treatment of decker effluents. However, for pine caustic extraction filtrate these residual salts can limit the reuse potential of the water. Second, color rejection in ultrafiltration is somewhat lower than that in reverse osmosis. The greater residual color in the effluent from an ultrafiltration system can be a limiting factor.

A review of the costs of commercially-available membrane equipment at the beginning of the project showed that spiral wound membranes would be the lowest in cost. Although some process limitations exist for membranes in this configuration, it was felt that this system would be adequate for processing both pine caustic extraction filtrate and decker effluents. The advantages and disadvantages of other membrane configurations have been discussed elsewhere (26). The two alternatives, hollow fine fiber and tubular configurations, are not thought to be attractive. The former is extremely susceptible to plugging by suspended solids, and the latter is a relatively high cost system. For these reasons the present study has focused solely on the use of membranes in the spiral wound configuration.

2. Need for Pilot Plant Studies

In a previous program sponsored by Champion Papers (unpublished) the necessity of conducting in-field pilot studies was apparent. In the preliminary program, several hundred gallon samples were shipped to pilot plant facilities for ultrafiltration tests. Data for membrane flux, membrane rejection efficiency, and material balances were obtained. However, factors which could not be evaluated were:

a) differences in treating "fresh" (minutes-old) and "aged" (days-old) feed materials;

b) pretreatment requirements for particulate removal to obtain stable long-term operation;

c) operability of a staged membrane system as a means of continuously producing a high-solids concentrate; that is, with feed "conversion" to permeate of $99^{+}\%$;

d) effective techniques for membrane cleaning as a means to sustain high-flux in long-term operation;

e) membrane flux and life in intermediate-term tests (six months). Note that long-term life tests were not a part of this program;

f) requirements for pH and temperature control, and performance at different pH, temperature, pressure and feed flow conditions;

g) examination of a statistically meaningful number of membrane modules to determine failure modes; and

h) system operability in the field.

Furthermore, feed pretreatment (filtration) data could only be obtained with fresh samples since particulate level and size were known to change with aging for pulp mill effluents.

Finally, variability in flow and composition of the wastes of interest, extremely important parameters in process design, had to be determined at the mill.

For these reasons, a pilot plant program was clearly warranted to realistically determine process technical and economic feasibilities.

3. Pine Caustic Extraction Filtrate; Flow Schematic and Typical Material Balance

Figure 2 shows a flow schematic for the treatment of pine caustic extraction filtrate by ultrafiltration. In the bleaching of pine pulp, the pulp flows through a series of stages. First is chlorination, with the aqueous waste discharged to drain. In the second stage, the pulp is extracted with caustic, and it is the effluent from this operation which contains the bulk of the color discharged from the bleaching system. The pulp is subsequently treated with calcium hypochlorite, and in other bleaching and washing stages.

One means of reducing the color discharge from a bleach plant is to reverse the hypochlorite and caustic extraction stages. Through this sequence a substantial fraction of the color is oxidized by hypochlorite, but at a cost of increased chemical usage.

In the proposed process, the traditional CEH ... bleaching sequence is used and the pine caustic extraction filtrate is processed by an ultrafiltration system (Figure 2). As described above, a membrane is selected which specifically concentrates the dissolved organics and color bodies. A purified, but not demineralized, water is obtained for reuse or disposal. Reuse would be limited to non-pulping operations because of the high chloride content. The organic concentrate is disposed of either by evaporation to approximately 50% solids, with subsequent ultimate disposal on land, or by incineration. A typical material balance for the ultrafiltration operation is shown in Figure 3.

By obtaining 90-96% color removal from the pine caustic extraction filtrate, approximately 50-60% of the total

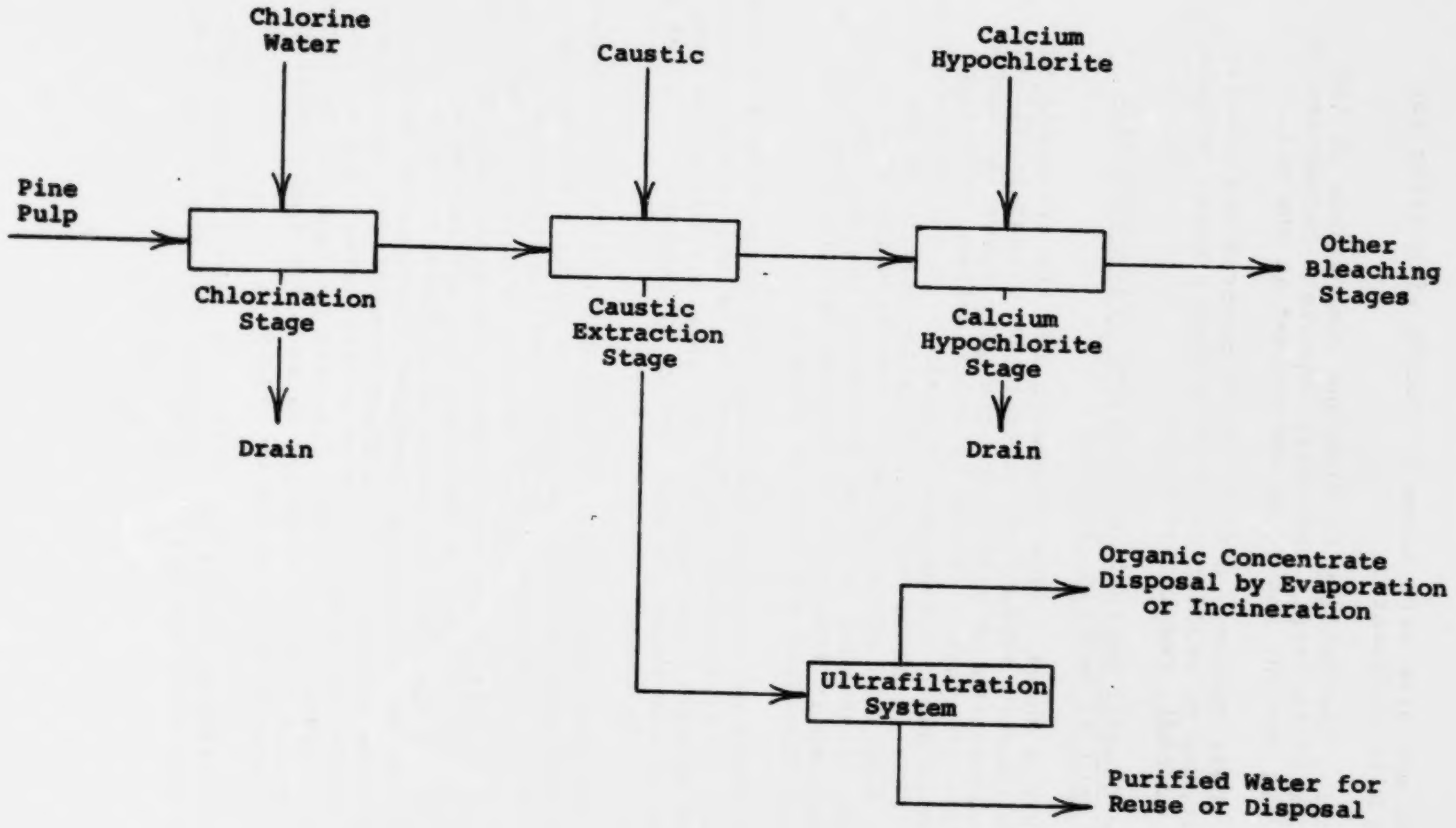

Chlorine
Water
Caustic
Calcium
Hypochlorite
Pine
Pulp
Other
Bleaching
Stages
Chlorination
Stage
Caustic
Extraction
Stage
Calcium
Hypochlorite
Stage
Drain
Drain
Organic Concentrate
Disposal by Evaporation
or Incineration
Ultrafiltration
System
Purified Water for
Reuse or Disposal

FIGURE 2: CAUSTIC EXTRACTION FILTRATE--FLOW SCHEMATIC

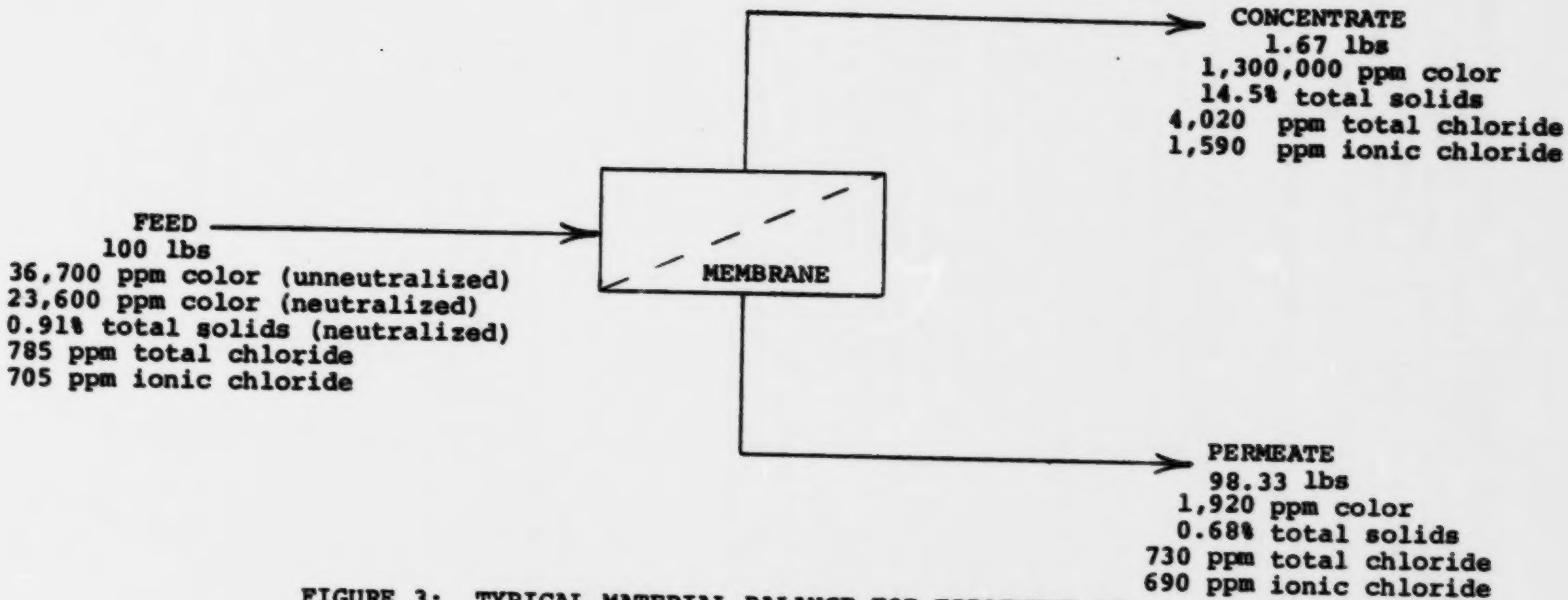

CONCENTRATE
1.67 lbs
1,300,000 ppm color
14.5% total solids
4,020 ppm total chloride
1,590 ppm ionic chloride
FEED
100 lbs
36,700 ppm color (unneutralized)
23,600 ppm color (neutralized)
0.91% total solids (neutralized)
785 ppm total chloride
705 ppm ionic chloride
MEMBRANE
PERMEATE
98.33 lbs
1,920 ppm color
0.68% total solids
730 ppm total chloride
690 ppm ionic chloride

FIGURE 3: TYPICAL MATERIAL BALANCE FOR TREATMENT OF CAUSTIC EXTRACTION FILTRATE BY ULTRAFILTRATION (Neutralization with H_2SO_4)

mill color will be removed. In addition, about 2 x 10^6 gpd of water will be available for potential reuse within the mill. If the water cannot be reused and is instead sewered to the waste treatment system, the organic loading (as B.O.D.) of the waste treatment system will be reduced by approximately 20%.

4. Decker Effluents: Flow Schematic and Typical Material Balance

The treatment of decker effluents is the other case of interest. While bleach plant effluents are found only in bleached kraft mills, decker effluent is found in all kraft mills. Thus, the treatment of decker effluent by ultrafiltration is an application of broader interest to the industry. Referring to Figure 4, wood chips are digested and the resulting pulp is transferred to a blow tank. The black liquor removed from the blow tank is processed in evaporators for chemical recovery. The pulp from the blow tank goes to a series of washers. At present, treated water is used in the washers, with the spent wash water processed in the black liquor recovery system. The pulp is then washed in a final decker operation, again with the addition of treated water. The decker effluent is too dilute to be processed with the black liquor, and is discharged to the waste treatment plant. At the North Carolina mill, approximately 2 x 10^6 gpd of treated water is added to the decker.

In the proposed process, the decker effluent is concentrated in an ultrafiltration system. The purified water is recycled to the final washer, or other pulp system uses, eliminating the need for adding treated water. This purified decker effluent could also be used at other points in the mill. The organic concentrate from the ultrafiltration system is processed in the black liquor recovery system. A typical material balance for the ultrafiltration operation is shown in Figure 5. Note that the color removal efficiency is better than for the pine caustic extraction filtrate case. This is because the color bodies are lower molecular weight for the latter, since substantial lignin fragmentation occurs in the bleach plant chlorination stage.

This treatment of decker effluent by ultrafiltration has several desirable features. These are

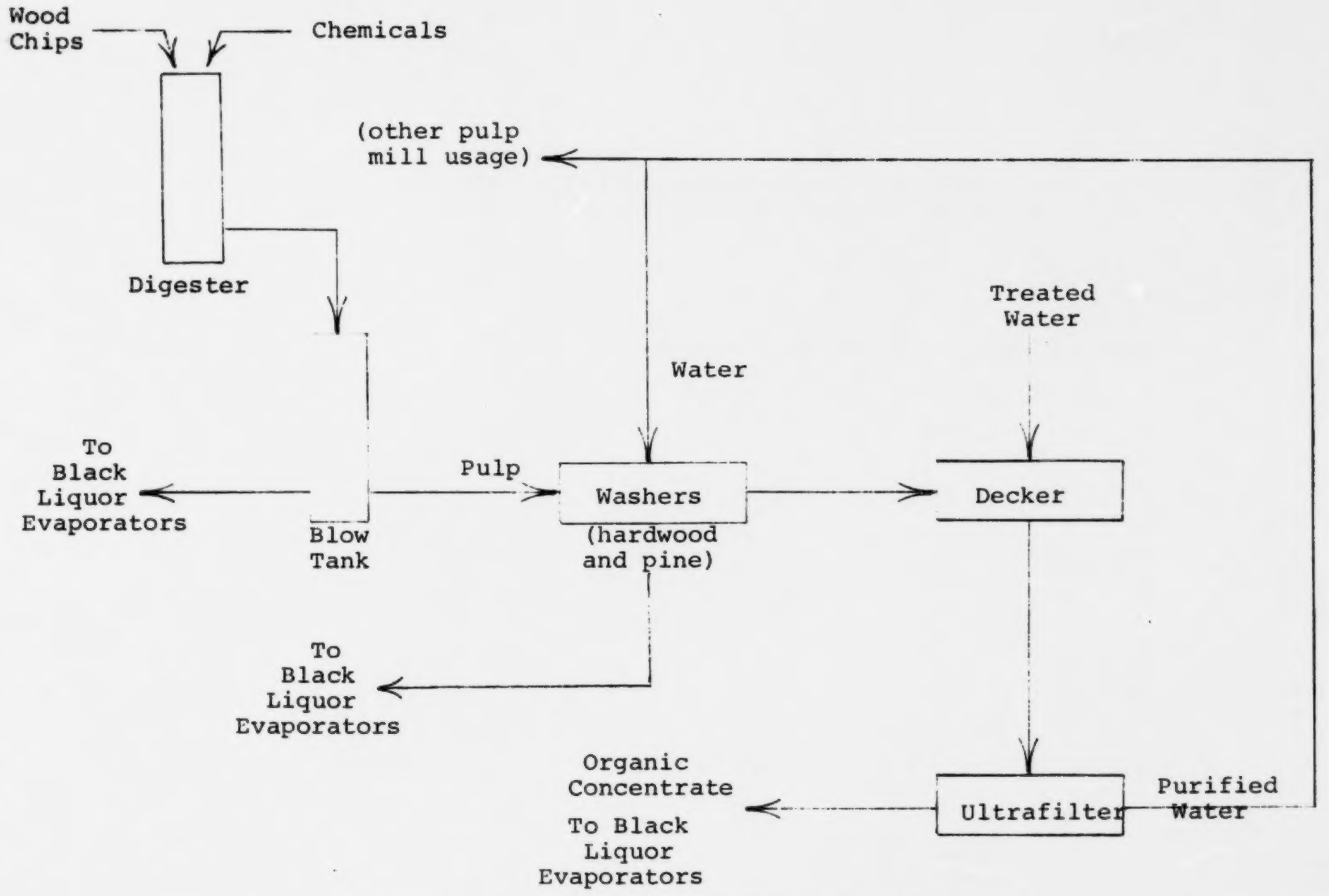

Wood Chips
Chemicals
Digester
(other pulp mill usage)
Blow Tank
To Black Liquor Evaporators
Pulp
Water
Washers
(hardwood and pine)
To Black Liquor Evaporators
Treated Water
Decker
Ultrafilter
Purified Water
Organic Concentrate
To Black Liquor Evaporators

FIGURE 4: DECKER EFFLUENTS--FLOW SCHEMATIC

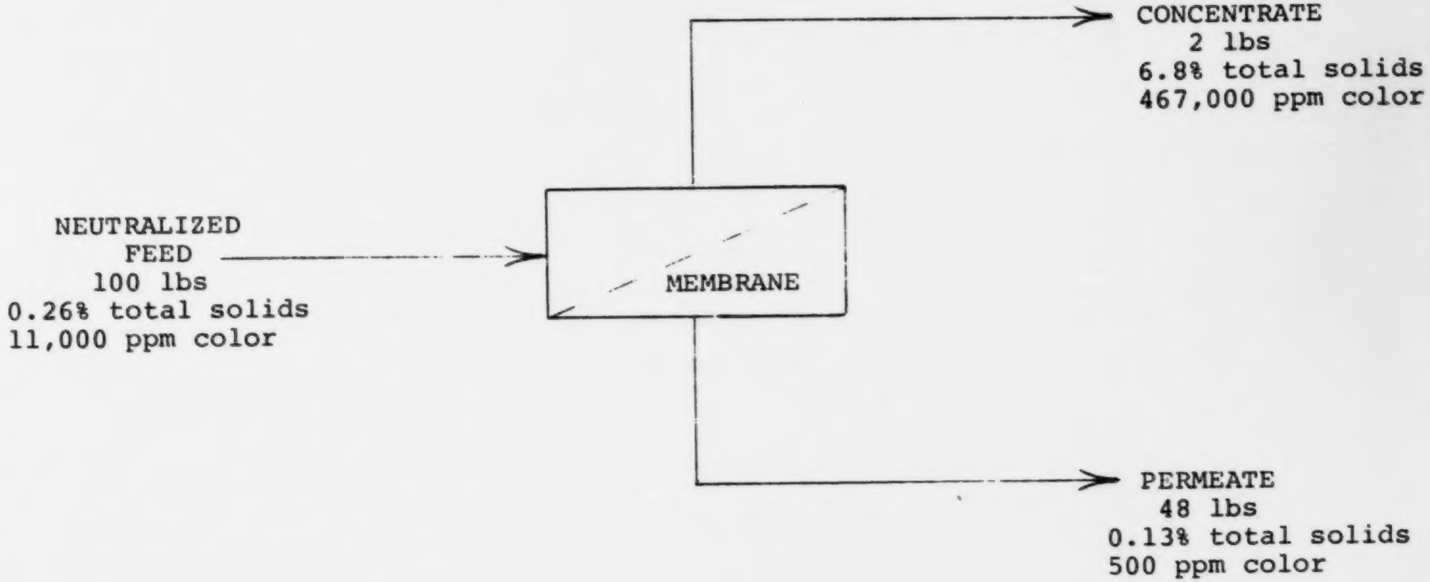

NEUTRALIZED
FEED
100 lbs
0.26% total solids
11,000 ppm color
MEMBRANE
CONCENTRATE
2 lbs
6.8% total solids
467,000 ppm color
PERMEATE
48 lbs
0.13% total solids
500 ppm color

FIGURE 5: TYPICAL MATERIAL BALANCE FOR TREATMENT OF HARDWOOD DECKER EFFLUENT BY ULTRAFILTRATION (Neutralization with H_2SO_4)

--the water usage, and subsequent treatment requirement in the waste treatment system, is reduced by 2 x 10^6 gpd;
--approximately 20% of the color in the total mill effluent is removed;
--salts ordinarily lost in the decker effluent are recovered and recycled to the black liquor evaporation system;
--organics ordinarily lost in the decker effluent are returned to the black liquor system and have a corresponding heating value; and
--the organic loading (as B.O.D.) to the existing waste treatment system is reduced by approximately 20%.

As described in detail below, the technical feasibility of treating both pine caustic extraction filtrate and decker effluents has been demonstrated. Projected costs for both cases are thought to be attractive. For the treatment of pine caustic extraction filtrate, ultrafiltration costs will be less than those for lime precipitation, activated carbon adsorption, or utilization of the alternative bleaching sequence. For the treatment of decker effluents, a positive return on investment might be realized.

SECTION IV

PILOT PLANT DESCRIPTION

A. GENERAL

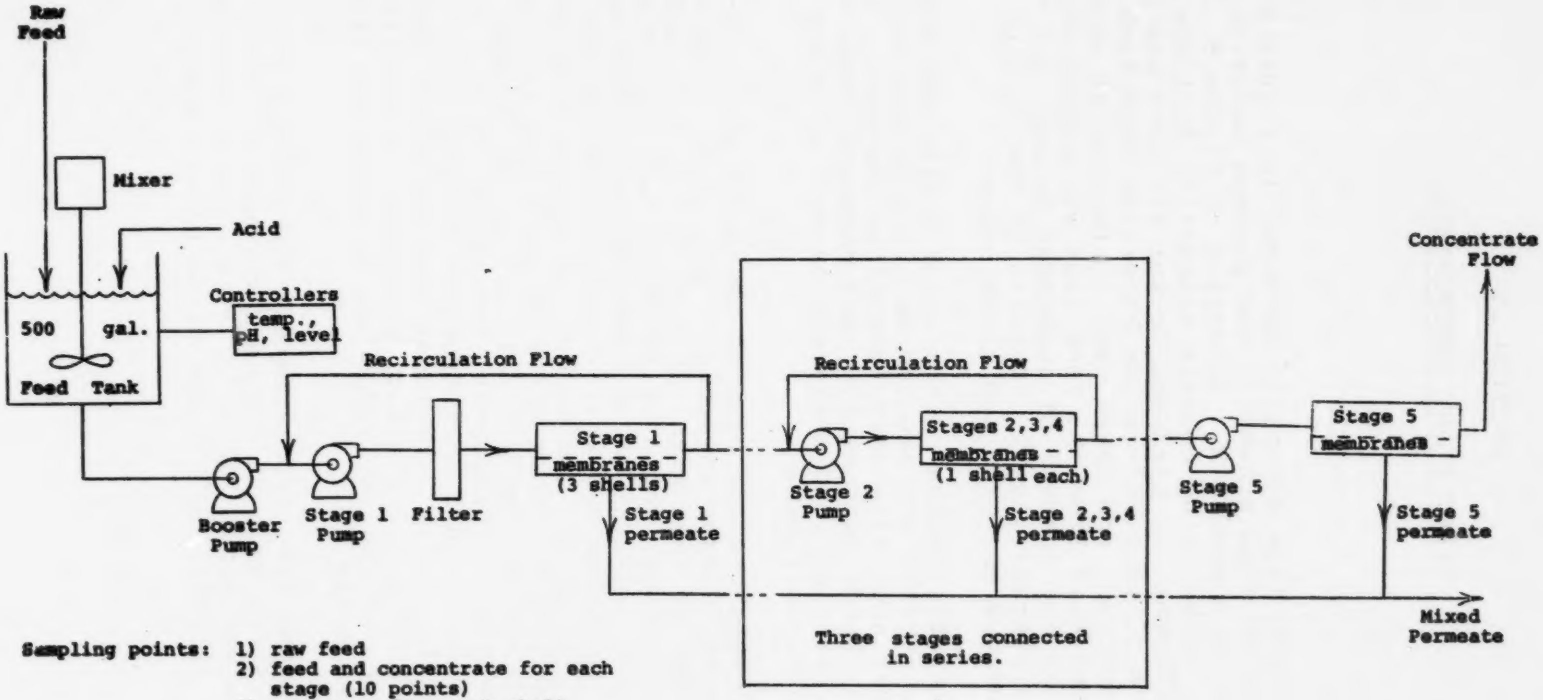
A generalized flow schematic is shown in Figure 6. A more detailed flow schematic and process description is contained in Appendix A. Referring to Figure 6, feed material, either pine caustic extraction filtrate, pine decker effluent, or hardwood decker effluent was piped from the mill to a 500-gallon Fiberglas feed tank. Temperature, pH, and level were controlled at this point. Neutralized feed from the feed tank was pumped through a filter(s) for removal of suspended solids, and then to the ultrafiltration unit. Details of the various filtration sequences employed are given below.

In the ultrafiltration system color bodies and organics were concentrated and removed as a low-volume concentrate, while water and salts were removed as permeate. During operation, feed, concentrate, and permeate flows were measured, sampled, and analyzed, as well as other intermediate process flows.

In the ultrafiltration part of the pilot plant, five recirculated "stages-in-series" were used. This design was selected so that high conversion with high color removal efficiency could be achieved (see Appendix A for details). Spiral wound membrane cartridges were installed in seven housings ("shells"); three shells were used in Stage 1; and one in each of Stages 2 through 5. A single shell held either three 24-inch long cartridges (T.J. Engineering, Downey, California; or Eastman Chemical Products, Kingsport, Tennessee) or two 36-inch long cartridges (Gulf Environmental Systems Co., San Diego, California). Cartridges from all three companies were used in the pilot plant operation. A detailed listing identifying cartridge use by stage and time is given in Appendix B.

Membrane areas were approximately 300 ft^2 for Stage 1, and 100 ft^2 for each of Stages 2 through 5. Total membrane area was about 700 ft^2. At a nominal membrane flux of 15 gal./day-ft^2 (gfd), the pilot plant capacity was 10,500 gpd.

Preceding page blank


Raw Feed
Mixer
Acid
Controllers
temp., pH, level
500 gal.
Feed Tank
Recirculation Flow
Booster Pump
Stage 1 Pump
Filter
Stage 1 membranes (3 shells)
Stage 1 permeate
Recirculation Flow
Stage 2 Pump
Stages 2,3,4 membranes (1 shell each)
Stage 2,3,4 permeate
Three stages connected in series.
Stage 5 Pump
Stage 5 membranes
Stage 5 permeate
Concentrate Flow
Mixed Permeate

Sampling points:
1) raw feed
2) feed and concentrate for each stage (10 points)
3) permeates from each shell (7 points)
4) mixed permeate

FIGURE 6. SIMPLIFIED PILOT PLANT FLOW SCHEMATIC

After pilot plant start-up in mid-August, 1972, it became apparent that the pre-filtration system provided with the pilot plant was inadequate for the removal of suspended solids. The specific manifestations of inadequate filtration were a rapid increase in pressure drop across the membrane cartridges, and a drastically reduced ultrafiltration rate with time. In addition, it was suspected that membrane fouling by colloidal and dissolved macromolecular materials was also a problem, although definitely of lesser importance than the flow-channel plugging created by particulates. Several changes were made to the pilot plant, including:

1. the installation of a single test cartridge which was used for tests to determine means of controlling the plugging and fouling problems;
2. the installation of more effective filters;
3. piping modifications to allow high-capacity, once-through water flushing of all stages; and
4. the installation of piping and tanks to allow detergent cleaning of membrane cartridges.

The sequence of changes can be summarized as follows:

Mid-August startup period: The originally installed Broughton 10μ basket filters were used.

Early September: A single cartridge was installed for special tests to examine the plugging/fouling problem. Also installed were a Bauer Hydrasieve filter for the removal of coarse fibers, and 1μ Cuno cartridge filters for more complete removal of suspended solids.

Mid-September: A Hydromation depth filter was put into use upstream of the Cuno polishing filters to prolong the polishing filter element life.

Mid October: A detergent cleaning system was integrated into the pilot plant. The Hydromation filter was replaced with a Shriver filter press to obtain increased capacity (prolonged filtration cycles).

End of November: Flush valves and the necessary piping for once-through flushing of all membrane shells was installed.

December: An automatic filter aid feeder was installed to permit unattended operation of the precoat filter over 24-hr periods.

Early February: The Shriver filter press was replaced by a Sparkler leaf filter to obtain more "representative" filtration data. A Sparkler Velmac disc filter was substituted for the Cuno polishing filters to examine a "backwashable" polishing filter.

Changes in the flow diagram that resulted from the equipment modifications are shown in detail in Appendix A (Figure A.2).

B. PRETREATMENT SEQUENCES

A more detailed flow schematic for the pretreatment operation is shown in Figure 7. Raw feed was piped from the mill to the 500-gallon feed tank. During part of the test period a screen (Bauer Hydrasieve) was used to remove coarse fibers and particulates from the raw feed prior to introduction into the feed tank.

As indicated, acid from a 55-gallon drum was pumped by an acid pump into the feed tank for neutralization. For the bulk of the program, sulfuric acid was used, but for a two-week period, hydrochloric acid was added.

When pre-coat filtration was employed, a continuous filter aid addition unit (BIF Industries screw-type adder) introduced filter aid directly into the feed tank, where vigorous mixing was achieved with a Lightnin mixer.

Neutralized, cooled feed was pumped by a filter pump through one of three filters, a Hydromation depth filter, a Shriver filter press, or a Sparkler leaf filter. The filtrate was held in a 100-gallon surge tank (two 55-gallon drums). In general, flow into this surge was through a float valve which kept the surge tank full. Thus, the filtrate flow was controlled by the subsequent rate of consumption in the pilot plant. At times excess filtrate was drawn off and either sewered or returned to the 500 gal. feed tank. When a precoat filter was being used, a precoat suspension was mixed in a 55 gal. drum connected to the suction of the filter pump. Filtrate was returned to the precoat mixing tank until an adequate precoat was developed.

Filtrate from the filtrate surge was pumped into the suction of the Goulds multistage Stage 1 pump. The

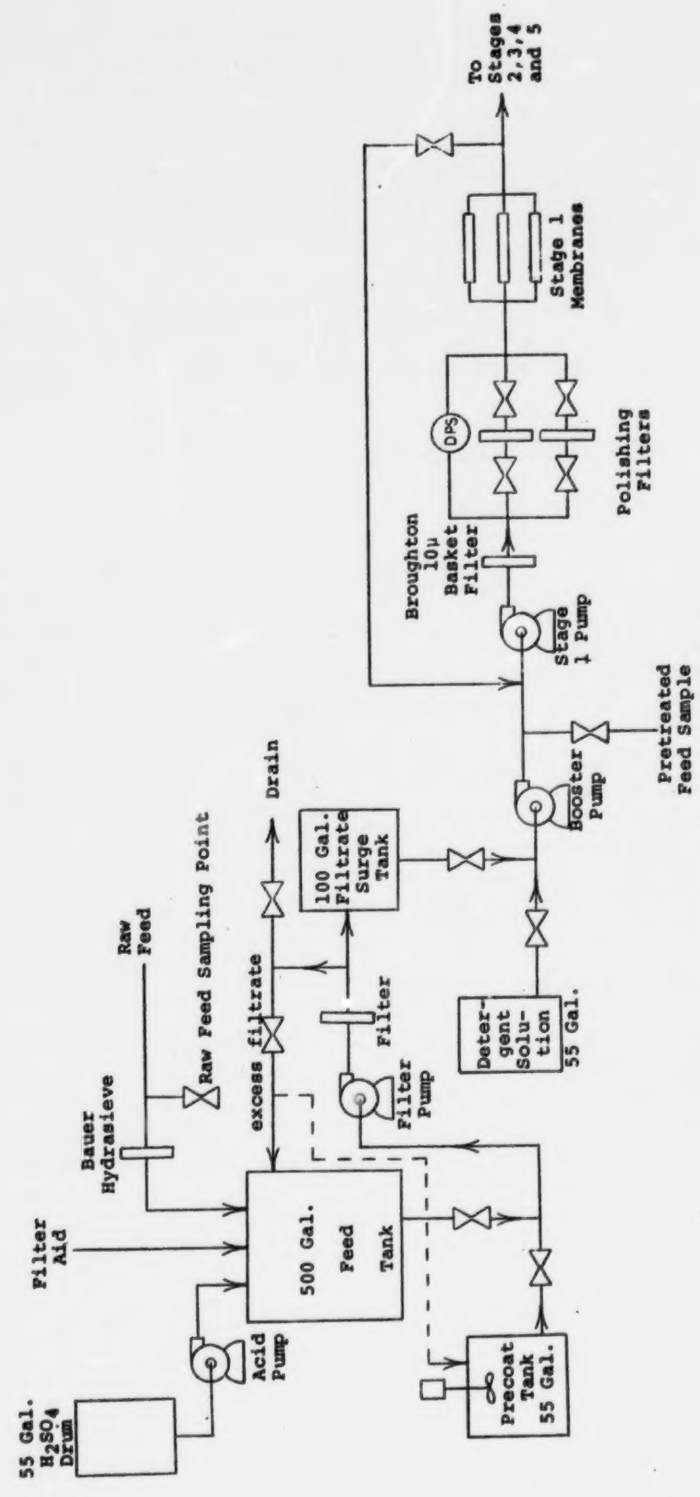

55 Gal.
H_2SO_4
Drum
Acid
Pump
Filter
Aid
Bauer
Hydrasieve
Raw
Feed
Raw Feed Sampling Point
500 Gal.
Feed
Tank
excess filtrate
Drain
Filter
Filter
Pump
100 Gal.
Filtrate
Surge
Tank
Precoat
Tank
55 Gal.
Deter-
gent
Solu-
tion
55 Gal.
Booster
Pump
Pretreated
Feed Sample
Stage
1 Pump
Broughton
10µ
Basket
Filter
DPS
Polishing
Filters
Stage 1
Membranes
To
Stages
2,3,4
and 5

FIGURE 7. FEED PRETREATMENT FLOW SCHEMATIC

outlet from the pump passed through two Broughton nominal 10μ basket filters. Flow was then through polishing filters (Cuno cartridges or Sparkler Velmac pot filter) to remove residual particulates. To avoid the buildup of an excessive pressure drop across the polishing filters, a differential pressure switch was installed. Excess pressure drop resulted in system shutdown. The final polished filtrate was introduced into the three parallel membrane shells of Stage 1.

A summary of the location in the process flow and the characteristics of the filters is given in Table 2. Manufacturer's information on the filters is contained in Appendix C.

C. OTHER SYSTEM MODIFICATIONS

1. Single Cartridge Tests

The single spiral wound cartridge was used for tests to characterize prefiltration efficiency. Raw feed was filtered by various means and processed in the single cartridge. The buildup in pressure drop across the cartridge and decrease in ultrafiltration rate were considered to reflect prefilter performance.

The single cartridge was connected in parallel to the Stage 1 shells, and fed by the Stage 1 pump (Appendix A, Figure 199). With this arrangement it was possible to operate the single cartridge simultaneously, or independently of, the pilot plant. The permeate and concentrate from the single cartridge were either sewered or returned to the 100 gal. filtrate surge tank.

2. Detergent Cleaning System

A 55-gal. barrel served as a mixing tank for cleaning solutions. The tank outlet was connected to the suction of the booster pump (Figure 7). When cleaning, the concentrate(s) and permeate(s) from the pilot plant were returned by hoses to the detergent solution tank, allowing the indefinite recirculation of detergent solution through the system. This eliminated the need to prepare large volumes of cleaning solutions.

3. Once-through Flushing

The original piping of the pilot plant did not permit

TABLE 2

FILTERS USED (IN ORDER OF FLOW SEQUENCE)

Filter	Location	Description	Function
1. Bauer Hydrasieve	in raw feed line to 500 gal. feed tank	Curved wire screen, 6 in. wide, 2 ft long, screen openings approx. 0.01 in. (see Fig. 8).	to remove coarse fibers and solids
2. Hydromation Depth Filter	after filter pump, before filtrate surge tanks	Granular PVC depth filter, with automatic backwashing. 1 ft^2 cross-sectional area.	fine filtration to remove particles of about 1μ and larger
3. Shriver Filter Press	"	24 plate filter press; 16 in. x 16 in. plates; total area of about 75 ft^2. Cloth used initially to hold filter aid; subsequently switched to paper sheets (see Fig. 14).	"
4. Sparkler Leaf Filter	"	Vertical leaf filter with 15.3 ft^2 area. Leaves with 316 ss wire mesh, covered with nylon bags, in carbon steel vessel. Contained high-pressure spray nozzles for cake removal (see Fig. 13).	"

TABLE 2 (continued)

FILTERS USED (IN ORDER OF FLOW SEQUENCE)

Filter	Location	Description	Function
5. Broughton Basket Filters	after Stage 1 pump, before Stage 1 membranes	Two model 3000 basket filters with nominal 10μ stainless steel mesh screens. Total area 5.6 ft^2. Included an automated differential-pressure-controlled backwashing sequence. A third basket filter (200 mesh screen) was used to filter water for backwashing (see Fig. 16).	to remove suspended solids of 10μ and larger (this was the initial filter provided with the pilot plant)
6. Cuno Cartridge Filters	after Broughton filter, before Stage 1 membranes	Two parallel filter housings, each containing two cartridges. Disposible 1-5μ cartridges in stainless steel housings. Area per filter of about 1.6 ft^2 for Micro-klean II elements and 1 ft^2 for Micro-Wynd II elements. Parallel filters permitted element change without system shutdown.	final polishing filtration before membrane elements
7. Sparkler Velmac Disc Filter	in place of Cuno filters	Contained backwashable polypropylene circular discs, 5μ retention, 15 ft^2 area.	"

once-through flushing of the membrane shells, i.e. the piping connected each stage to the next. It was decided that cleanup of plugged membrane cartridges could be facilitated by once-through flushing with water. Consequently, a water line was connected to the suction of the pump for each stage, and flush valves were piped into the concentrate lines from each stage. This arrangement permitted once-through flushing of each shell in a "forward-flow" direction. A subsequent piping modification allowed once-through "reverse-flow" flushing.

Photographs of various system components are contained in Figures 8-21.

D. DESCRIPTION OF MEMBRANE CARTRIDGES

The spiral wound cartridges consist of a membrane envelope and mesh spacer that have been rolled around a PVC tube (see Figure 22). One or more cartridges in series can be installed in a housing, which serves as a pressure vessel. The process fluid passes through the open space provided by the mesh spacer. The permeate produced spirals through the porous backing inside the membrane envelope into the PVC center tube. A seal between the OD of the cartridge and the ID of the shell prevents the process liquid from bypassing the cartridge.

For the pilot plant program, 7-ft long, 4-in diameter epoxy-coated steel shells were used. For the special tests with a single cartridge, a 30-in long, 4-in diameter PVC shell was employed.

Four different types of cartridges were purchased from three different suppliers. All membranes were cellulose acetate. More specific information on the cartridges can be found in Table 3.

The Gulf cartridges had the highest salt rejection and lowest flux. These were therefore used primarily in Stages 4 and 5, where the greatest loss of color into the permeate could occur and where the lowest flux was expected due to high feed concentrations. One set of three cartridges with corrugated flow channel spacers was purchased for the purpose of comparing this type of spacer with the standard mesh spacer. The former is less susceptible to plugging by suspended solids.

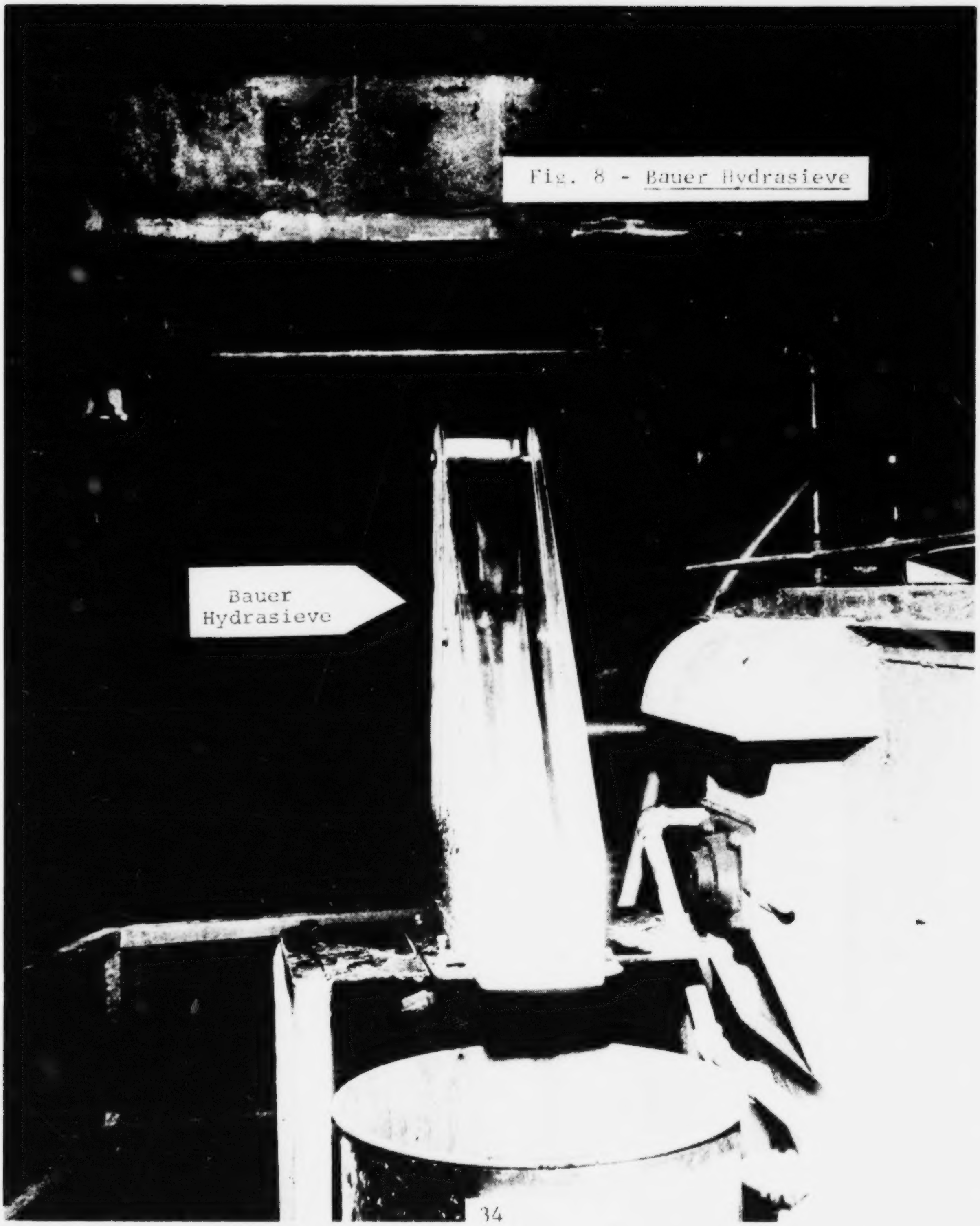


Fig. 8 - Bauer Hydrasieve

Bauer
Hydrasieve

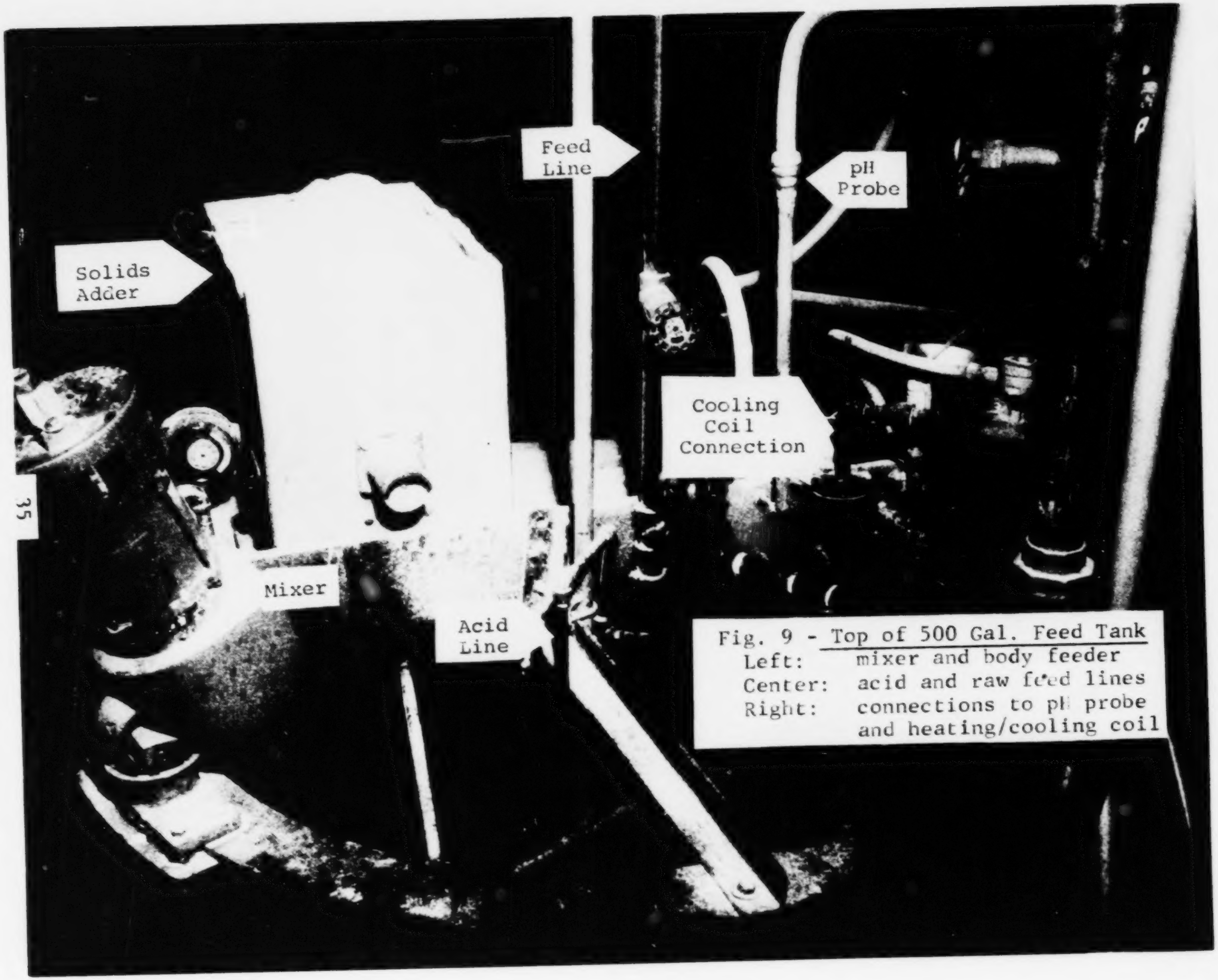

Feed Line
pH Probe
Solids Adder
Cooling Coil Connection
Mixer
Acid Line

Fig. 9 - Top of 500 Gal. Feed Tank
Left: mixer and body feeder
Center: acid and raw feed lines
Right: connections to pH probe and heating/cooling coil

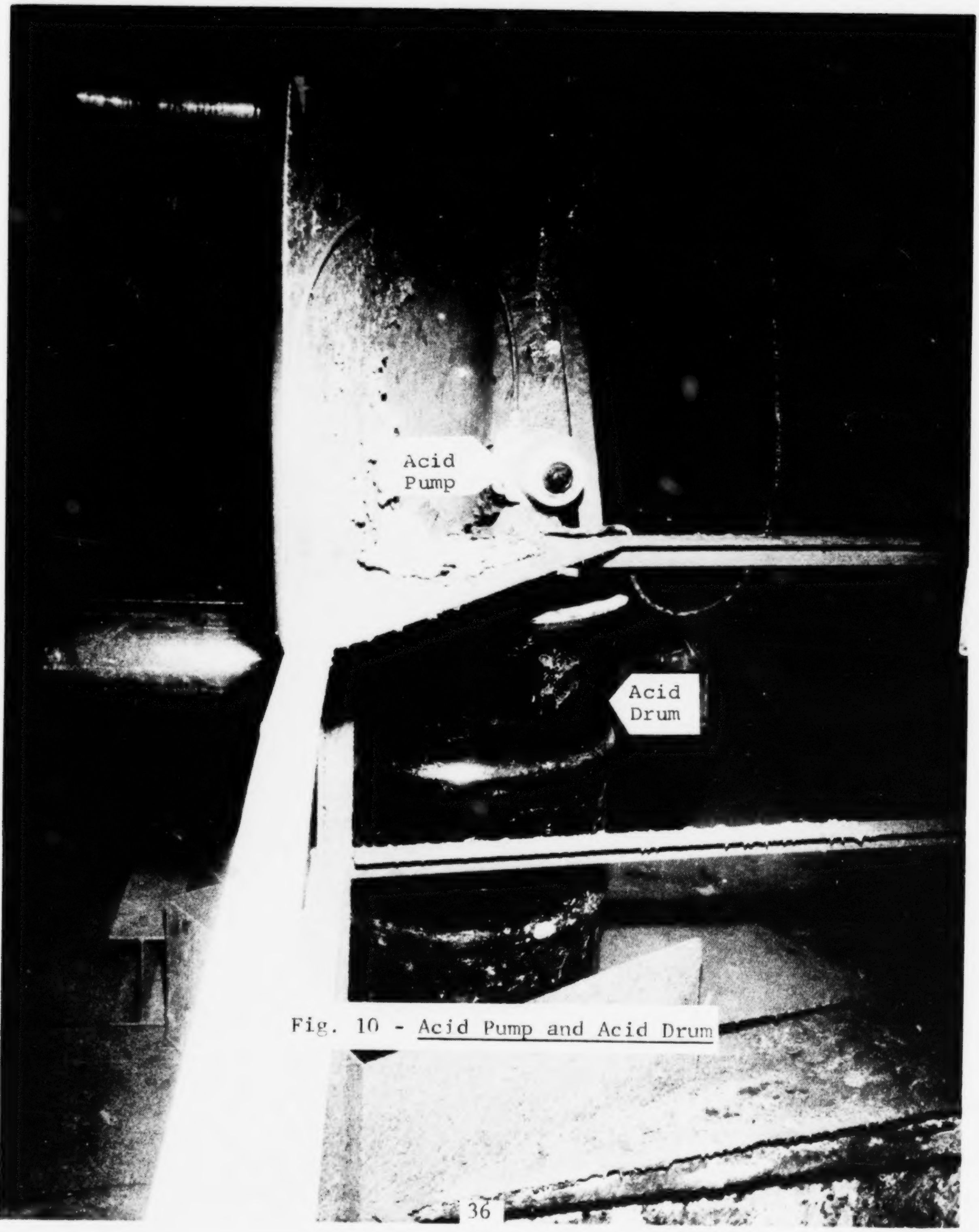

Acid
Pump
Acid
Drum

Fig. 10 - Acid Pump and Acid Drum

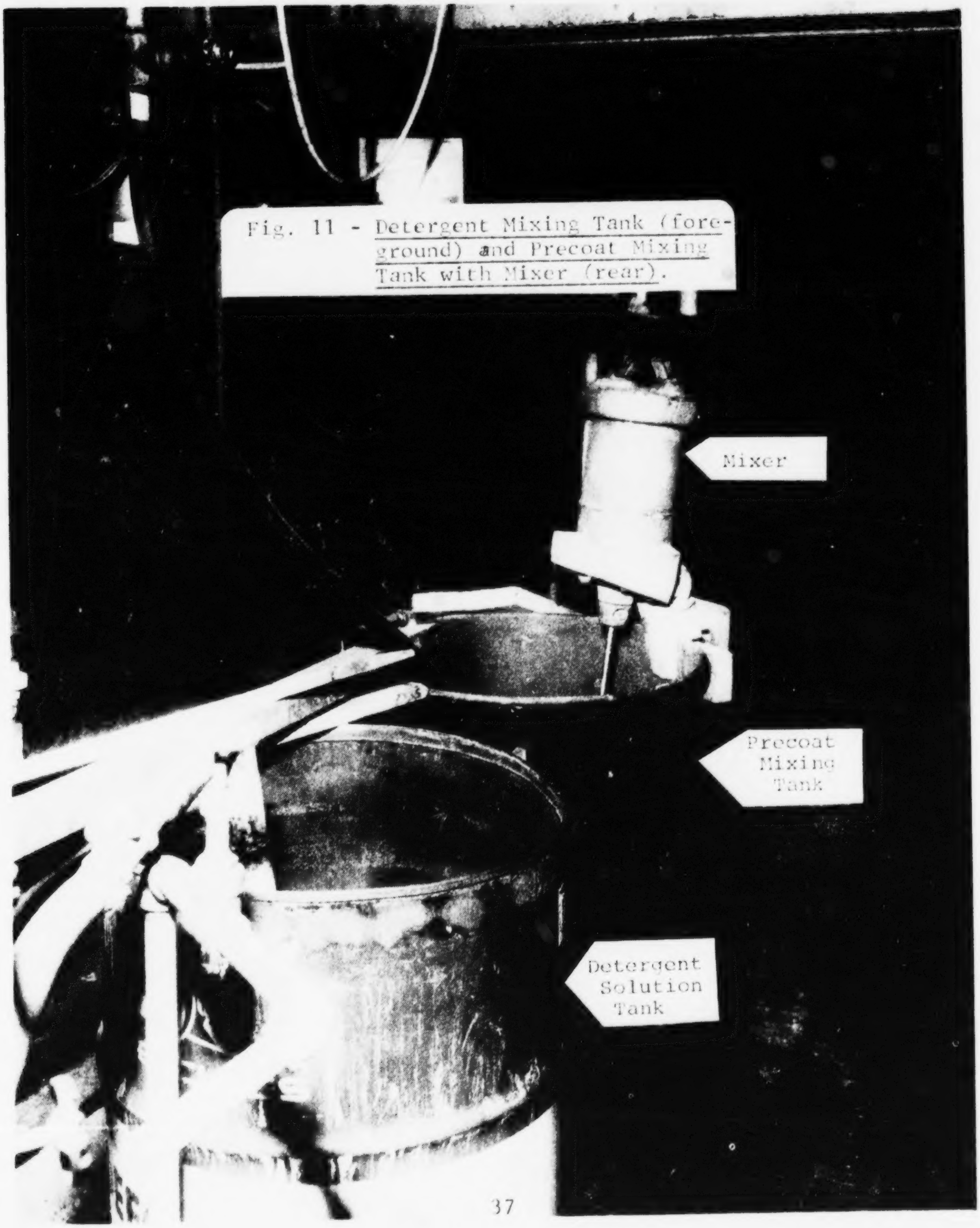

Mixer
Precoat Mixing Tank
Detergent Solution Tank

Fig. 11 - Detergent Mixing Tank (foreground) and Precoat Mixing Tank with Mixer (rear).

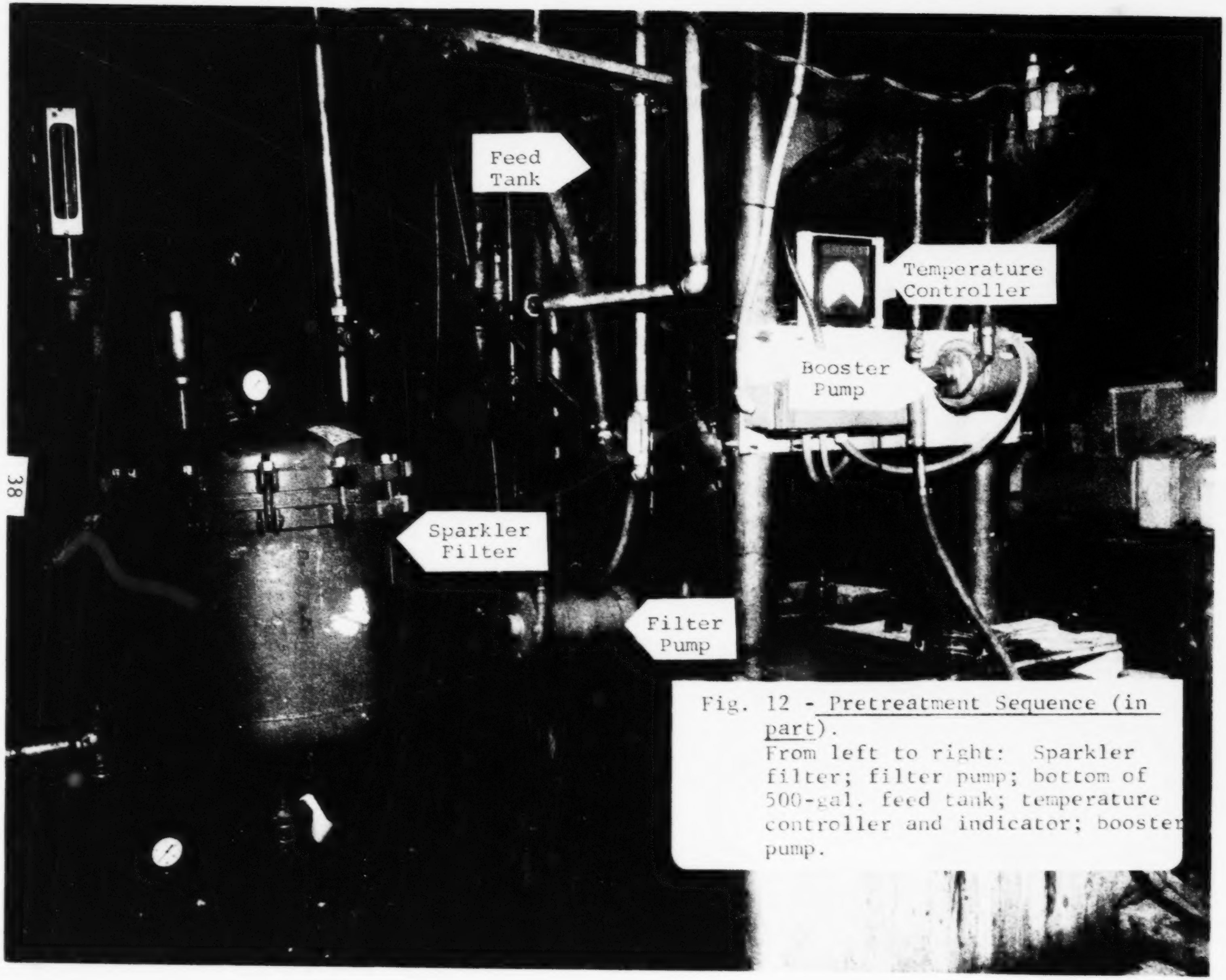

Feed Tank
Temperature Controller
Booster Pump
Sparkler Filter
Filter Pump

Fig. 12 - Pretreatment Sequence (in part).
From left to right: Sparkler filter; filter pump; bottom of 500-gal. feed tank; temperature controller and indicator; booster pump.

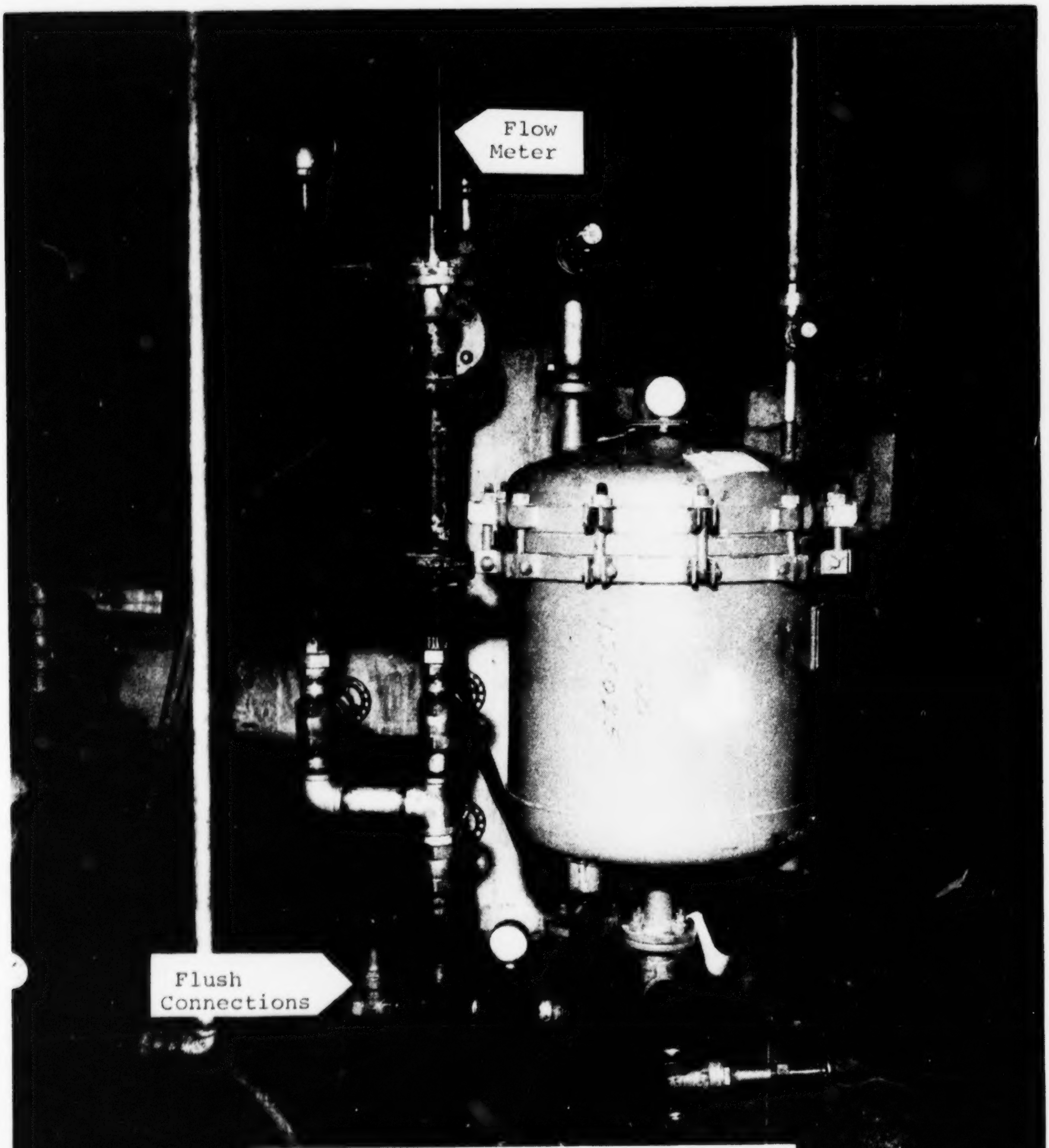

Flow Meter
Flush Connections

Fig. 13 - Details of Sparkler Filter
Pressure gauges, filtrate flow meter, flush connections.

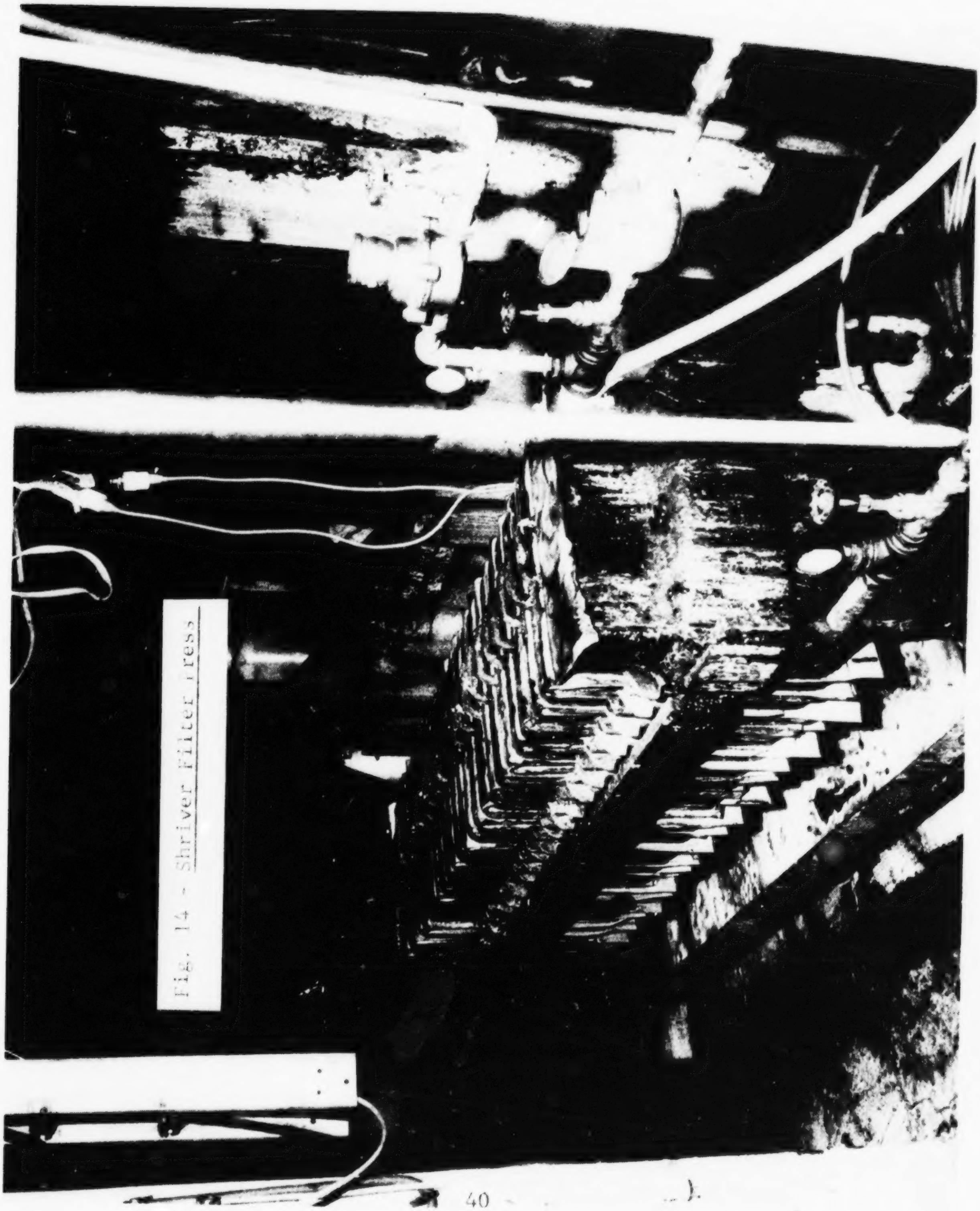


Fig. 14 - Shriver Filter Press

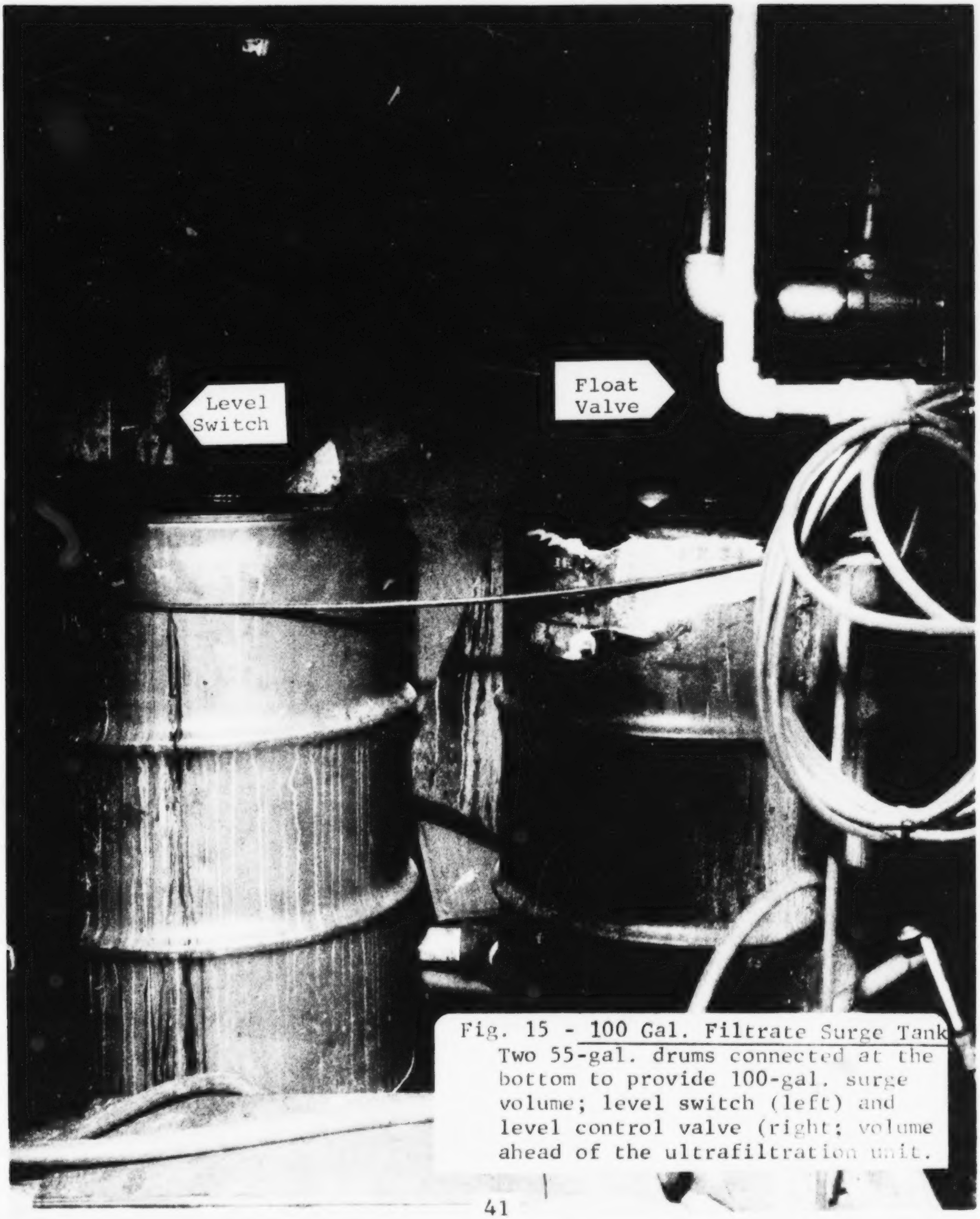

Level Switch
Float Valve

Fig. 15 - 100 Gal. Filtrate Surge Tank
Two 55-gal. drums connected at the bottom to provide 100-gal. surge volume; level switch (left) and level control valve (right; volume ahead of the ultrafiltration unit.

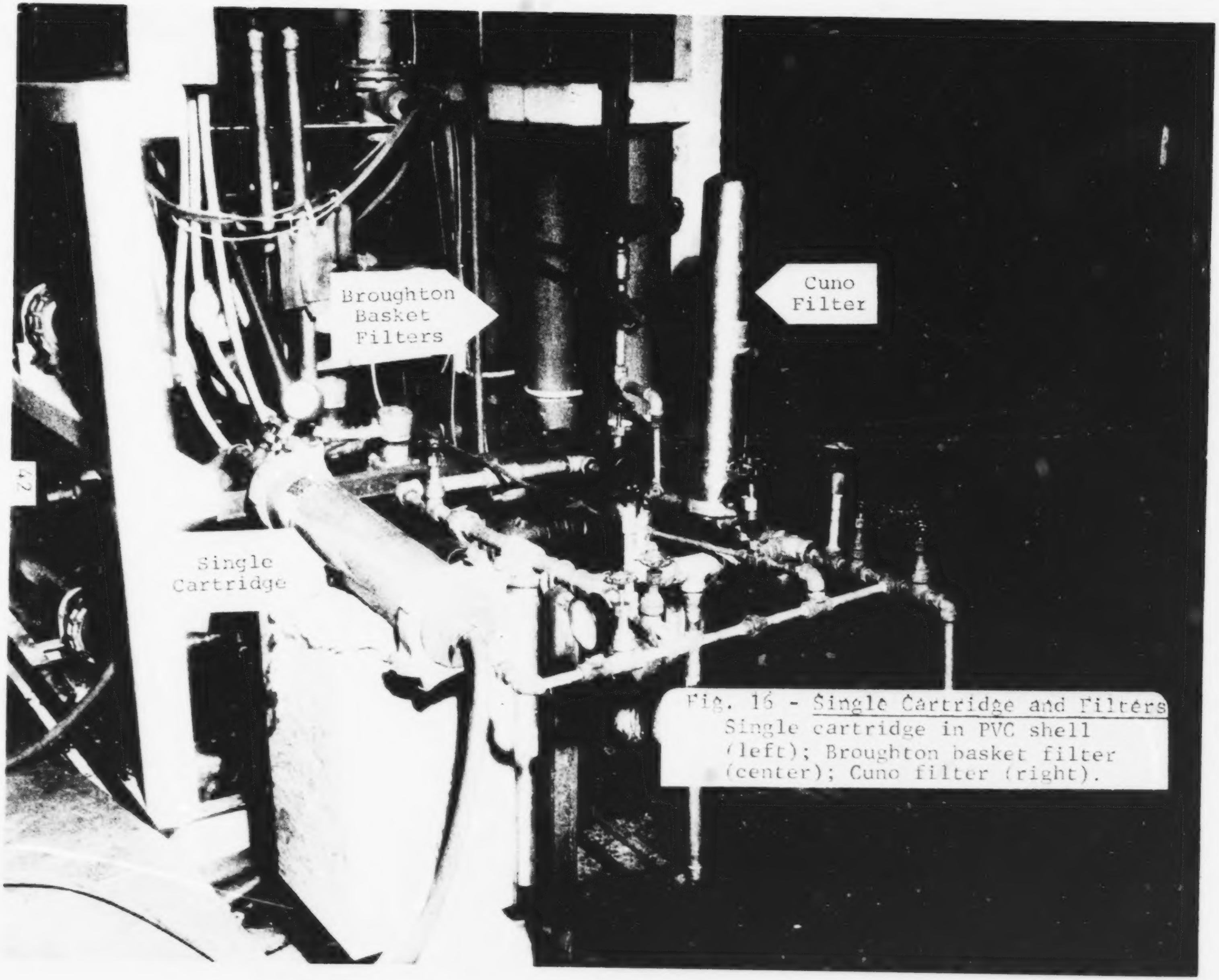

Broughton
Basket
Filters
Cuno
Filter
Single
Cartridge

Fig. 16 - Single Cartridge and Filters
Single cartridge in PVC shell (left); Broughton basket filter (center); Cuno filter (right).

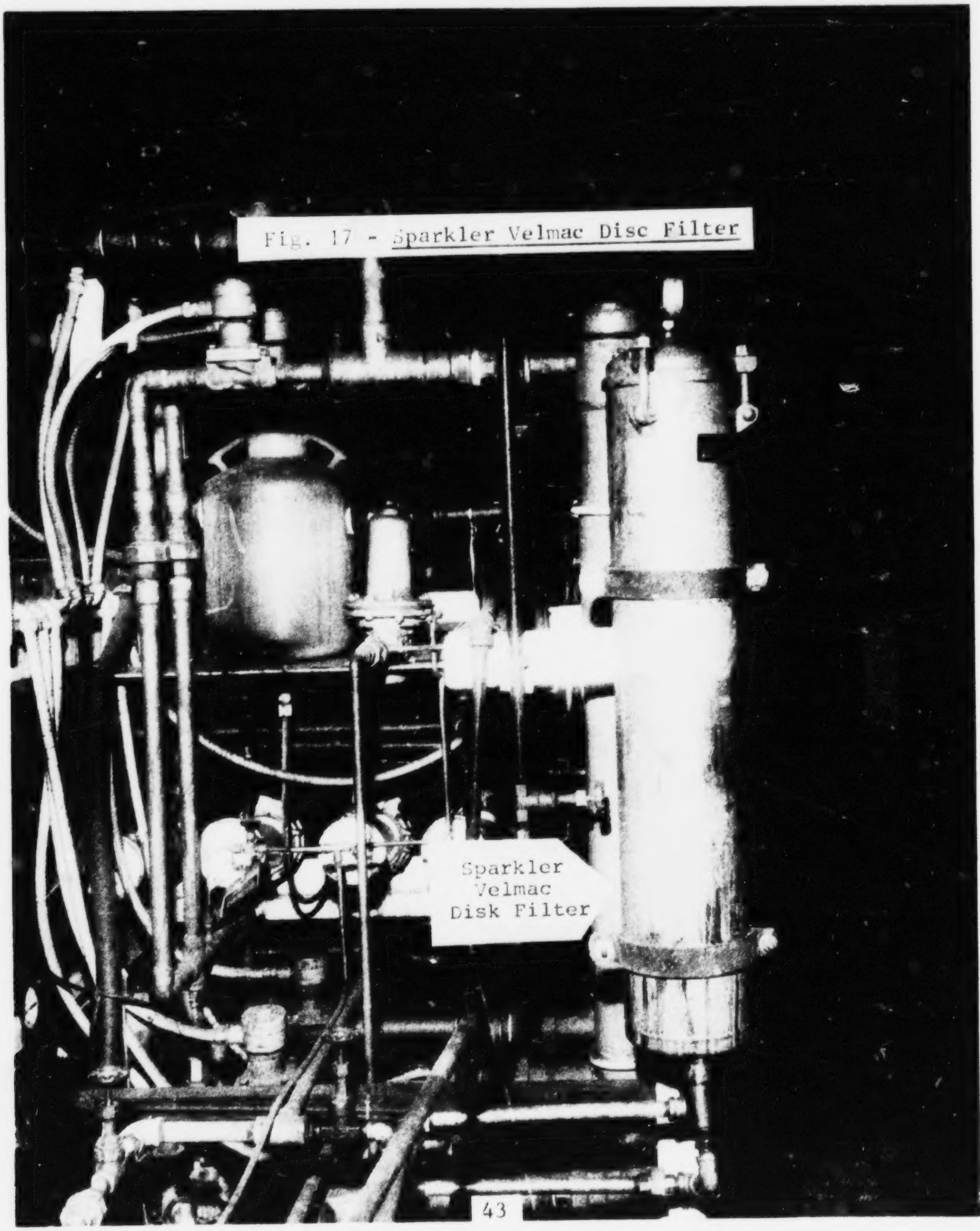


Fig. 17 - Sparkler Velmac Disc Filter

Sparkler
Velmac
Disk Filter

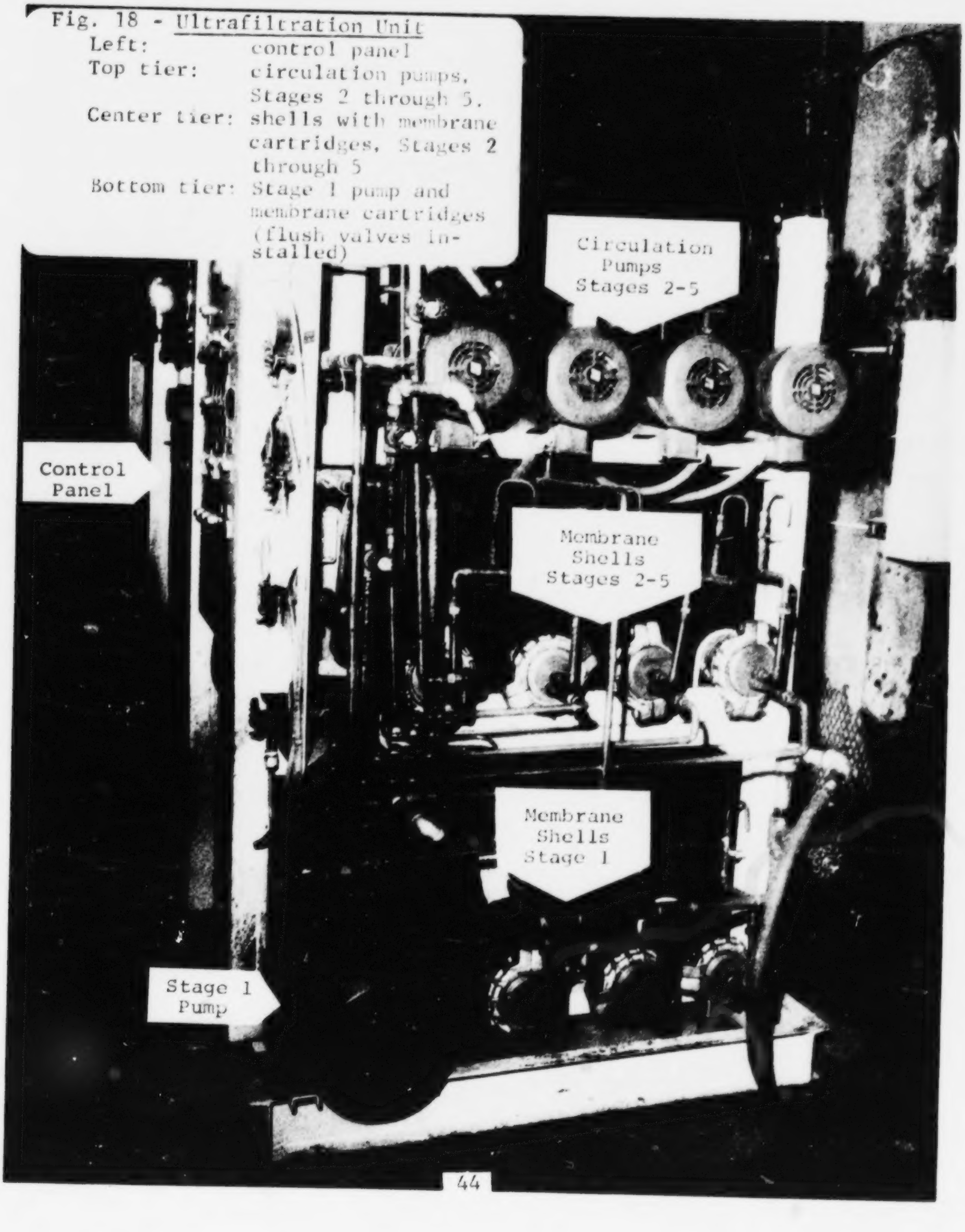

Circulation Pumps Stages 2-5
Control Panel
Membrane Shells Stages 2-5
Membrane Shells Stage 1
Stage 1 Pump

Fig. 18 - Ultrafiltration Unit

Left: control panel

Top tier: circulation pumps, Stages 2 through 5.

Center tier: shells with membrane cartridges, Stages 2 through 5

Bottom tier: Stage 1 pump and membrane cartridges (flush valves installed)

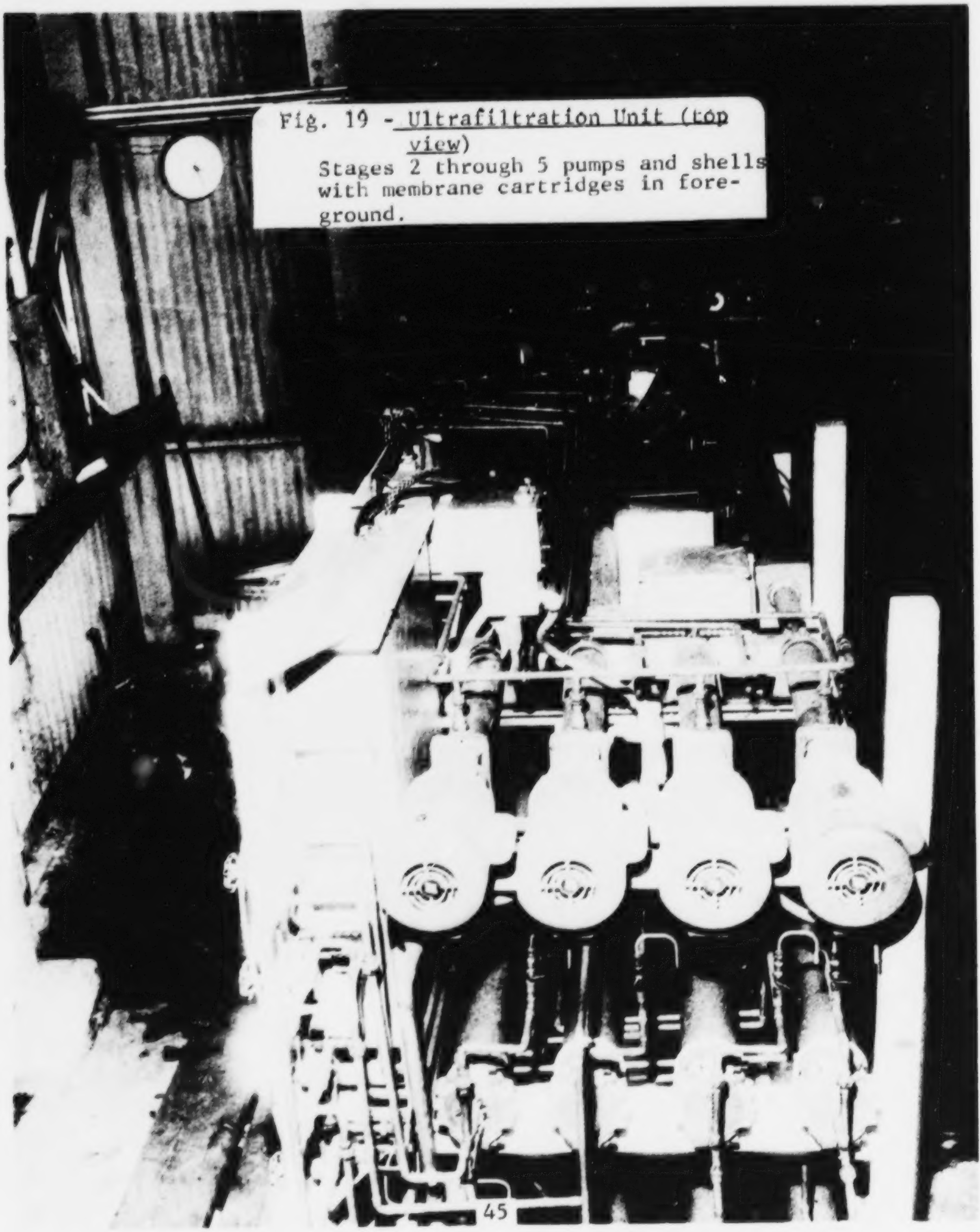


Fig. 19 - Ultrafiltration Unit (top view)
Stages 2 through 5 pumps and shells with membrane cartridges in foreground.

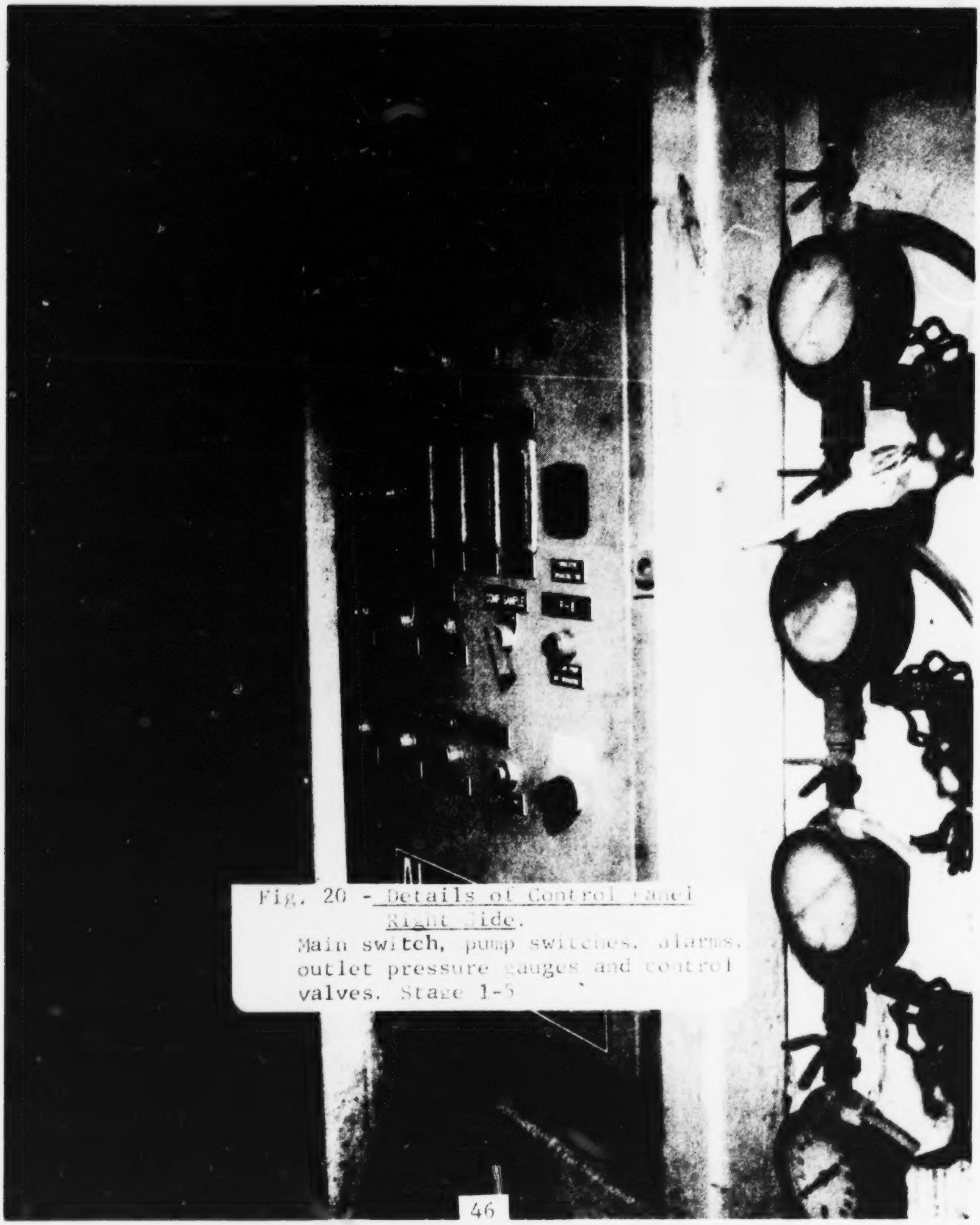


Fig. 20 - Details of Control Panel Right Side.

Main switch, pump switches, alarms, outlet pressure gauges and control valves. Stage 1-5

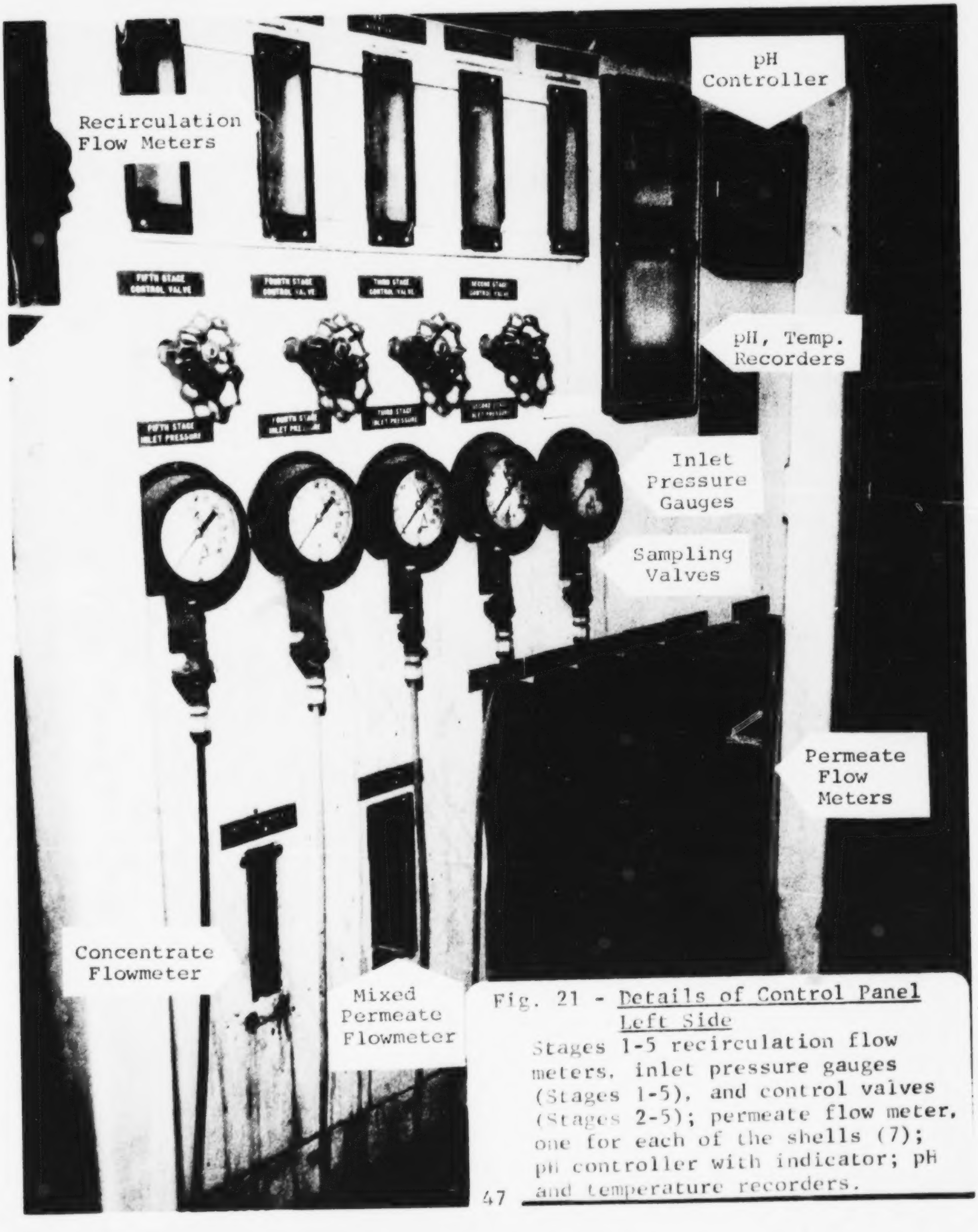
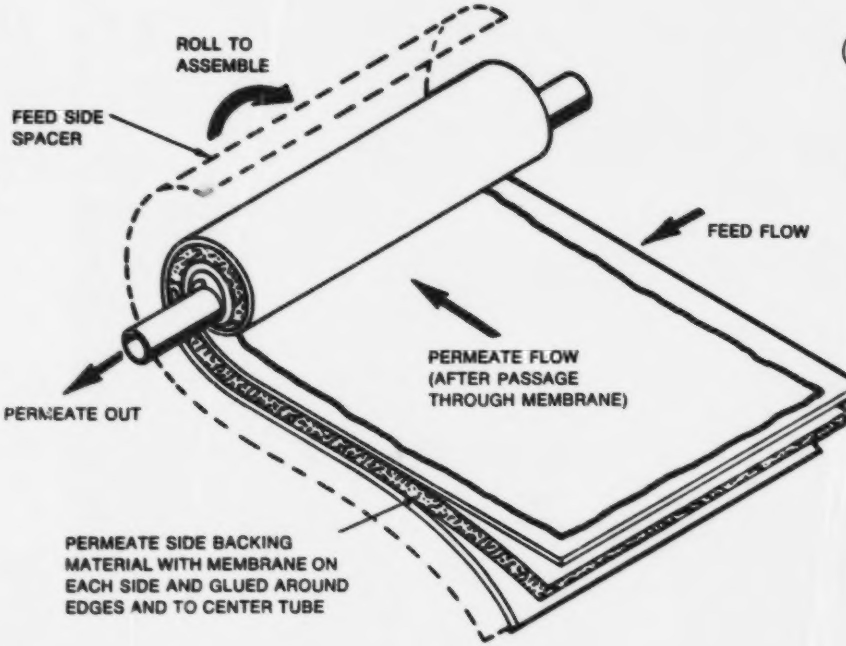
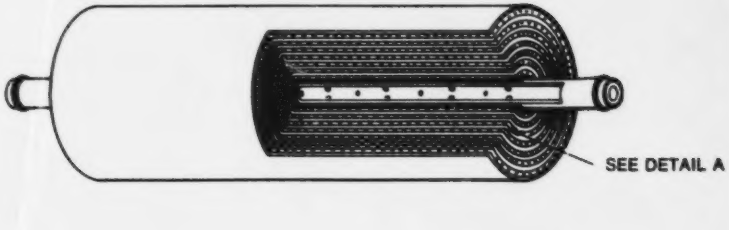

pH Controller
Recirculation Flow Meters
FIFTH STAGE CONTROL VALVE
FOURTH STAGE CONTROL VALVE
THIRD STAGE CONTROL VALVE
SECOND STAGE CONTROL VALVE
pH, Temp. Recorders
FIFTH STAGE INLET PRESSURE
FOURTH STAGE INLET PRESSURE
THIRD STAGE INLET PRESSURE
SECOND STAGE INLET PRESSURE
Inlet Pressure Gauges
Sampling Valves
Permeate Flow Meters
Concentrate Flowmeter
Mixed Permeate Flowmeter

Fig. 21 - Details of Control Panel Left Side

Stages 1-5 recirculation flow meters, inlet pressure gauges (Stages 1-5), and control valves (Stages 2-5); permeate flow meter, one for each of the shells (7); pH controller with indicator; pH and temperature recorders.


ROLL TO ASSEMBLE
FEED SIDE SPACER
FEED FLOW
PERMEATE OUT
PERMEATE FLOW (AFTER PASSAGE THROUGH MEMBRANE)
PERMEATE SIDE BACKING MATERIAL WITH MEMBRANE ON EACH SIDE AND GLUED AROUND EDGES AND TO CENTER TUBE


SEE DETAIL A

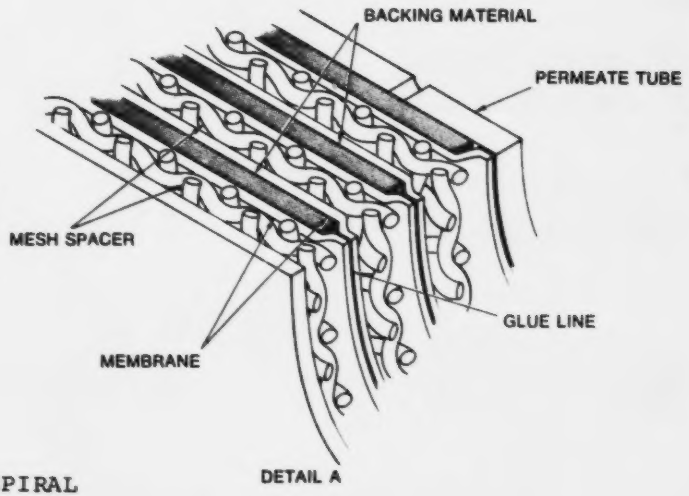

BACKING MATERIAL
PERMEATE TUBE
MESH SPACER
GLUE LINE
MEMBRANE
DETAIL A

FIGURE 22. SPIRAL WOUND MODULE DESIGN

TABLE 3

CHARACTERISTICS OF MEMBRANE CARTRIDGES

SUPPLIER	Gulf Environmental Systems	TJ Engineering	TJ Engineering	Eastman Chemical Products
model no.	4000	UF-H-32	UF	MK 00 A
membrane type	UF	Eastman HT 00	Eastman HT 00	HT 00
spacer material	mesh	mesh	corrugated	mesh
length of cartridge, inches	36	24	24	24
number per shell	2	3	3	3
membrane area per cartridge, ft^2	50	32-35	12-15	32-35
nominal salt rejection, %	approx. 30	0-10	0-10	0-10
nominal flux, gfd at 100 psig, 70° F.	7-10	35-50	35-50	35-50
number purchased	4	45	3	3
number of brine seals	1	2	2	1
brine sealing point during normal operation	upstream	downstream	downstream	upstream

E. SAMPLING AND ANALYTICAL PROCEDURES

Referring to Figures 6 and 7, the following samples were collected:

- --raw (untreated) feed;
- --neutralized and filtered feed (feed to Stage 1);
- --final concentrate (Stage 5 concentrate);
- --mixed permeate (from all 5 stages);
- --feed samples for all stages (5); and
- --permeate samples for all stages (5).

Analytical procedures are detailed in Table 4. The analyses for pH, total solids, suspended solids and color of the raw and pretreated feed, final concentrate and mixed permeate were performed on a regular basis. Occasionally, analyses of feed and permeate samples from individual membrane stages were also carried out. A few samples were analyzed for other constituents (ash, various ions, and suspended solids composition and particle size distribution) to better characterize performance of the system.

Suspended solids analyses were performed by the standard gravimetric method. However, it was found that only relatively small samples, 50-100 ml, could be filtered through the 0.45μ Millipore membrane filters before blinding occurred. Thus, the suspended solids analyses were only approximate, and poorly reproducible, since the total amounts of suspended solids present in the samples were small, and difficult to measure accurately.

A turbidimeter and a nepholometer were also tried to assay for suspended solids, but both instruments were found to lack sensitivity because of strong light absorption by the highly colored samples.

TABLE 4

ANALYTICAL PROCEDURES

Analysis For	Procedure *
pH	Standard pH meter
Color, Cobalt units	Absorbance at 465 mμ compared to absorbance of standard Pt/Co. solution, No. 118 at pH 7.6, "Field" color measured at as-is pH.
Total Solids	Gravimetric, No. 148A
Total Dissolved Solids	Gravimetric, No. 148B
Suspended Solids	Gravimetric, No. 148C
Ash	Gravimetric, No. 148D
Ionic Chloride	Calibrated chloride ion electrode, No. 203C
Na+	Flame Photometric Method, No. 153A
Ca++	EDTA Titrimetric, No. 67C
So =	Gravimetric, No. 238A
Fe+++	Colorimetric, No. 144B
Particle Size Distribution of Suspended Solids	Sizing on filters fllowed by analysis of fraction passing through 44μ filter with Coulter counter.

*Referenced number is the method in Standard Methods for Examination of Water and Wastewater, APHA, 13th Edition, 1971.

SECTION V

RESULTS AND DISCUSSION

A. FEED PRETREATMENT AND CHARACTERISTICS

The pilot plant was operated on pretreated pine caustic extraction filtrate for most of the test program, and on hardwood and pine decker effluents for a limited period during January, 1973.

Before introduction into the ultrafiltration section of the pilot plant all three effluents were neutralized to a pH of 6 to 7, cooled to a temperature of 95 to 105°F, and filtered to remove suspended solids. A description of the pretreatment section of the pilot plant was given in Section III.B. Results pertinent to the evaluation of the pretreatment operations are discussed below.

1. Temperature

Cellulose acetate ultrafiltration membranes show accelerated hydrolysis and compaction (irreversible loss of flux due to collapse of the porous membrane structure) with increasing temperature, and especially when exposed to temperatures in excess of 110°F. All three effluents treated in the pilot plant are generally discharged at a temperature of 120-135°F, depending on mill operating conditions and the season. Therefore, it was necessary to cool the feed, which was done by passing cold water through the coil heat exchanger in the feed tank. In the course of the experimental program, the operating temperature was not intentionally varied, and most data are for operation at a feed temperature between 95 and 105°F.

2. pH

Cellulose acetate membranes show satisfactory life when operated within a pH range of 3 to 7. All three effluents, however, are highly alkaline, with pH's ranging from pH 10 to pH 12. Therefore, the effluents were neutralized to a pH of 6 to 7, by addition of sulfuric acid. Figures 23 to 25 contain pH curves for the neutralization of pine caustic extraction filtrate, pine decker

Preceding page blank

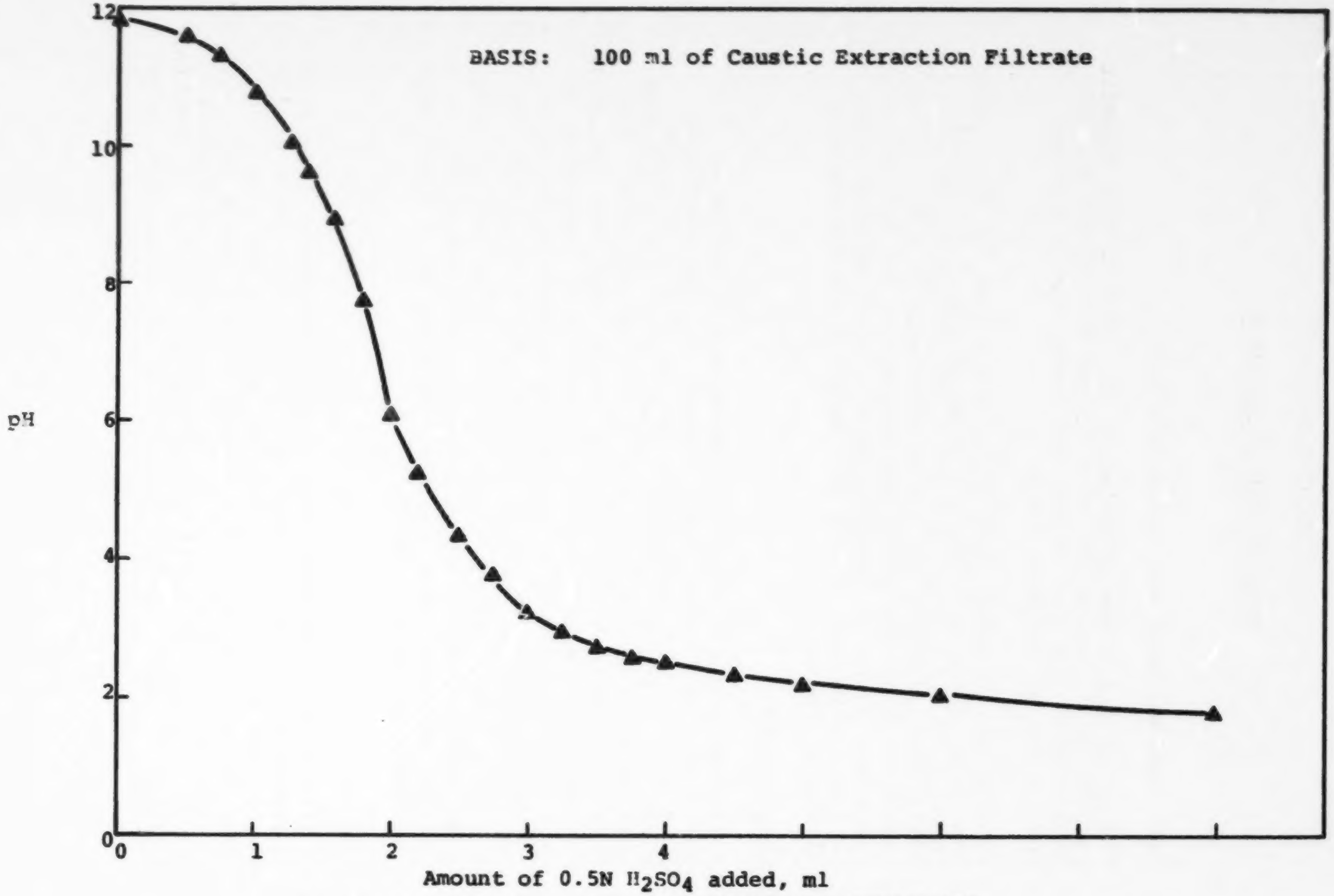


FIGURE 23: SULFURIC ACID REQUIREMENT TO NEUTRALIZE CAUSTIC EXTRACTION FILTRATE

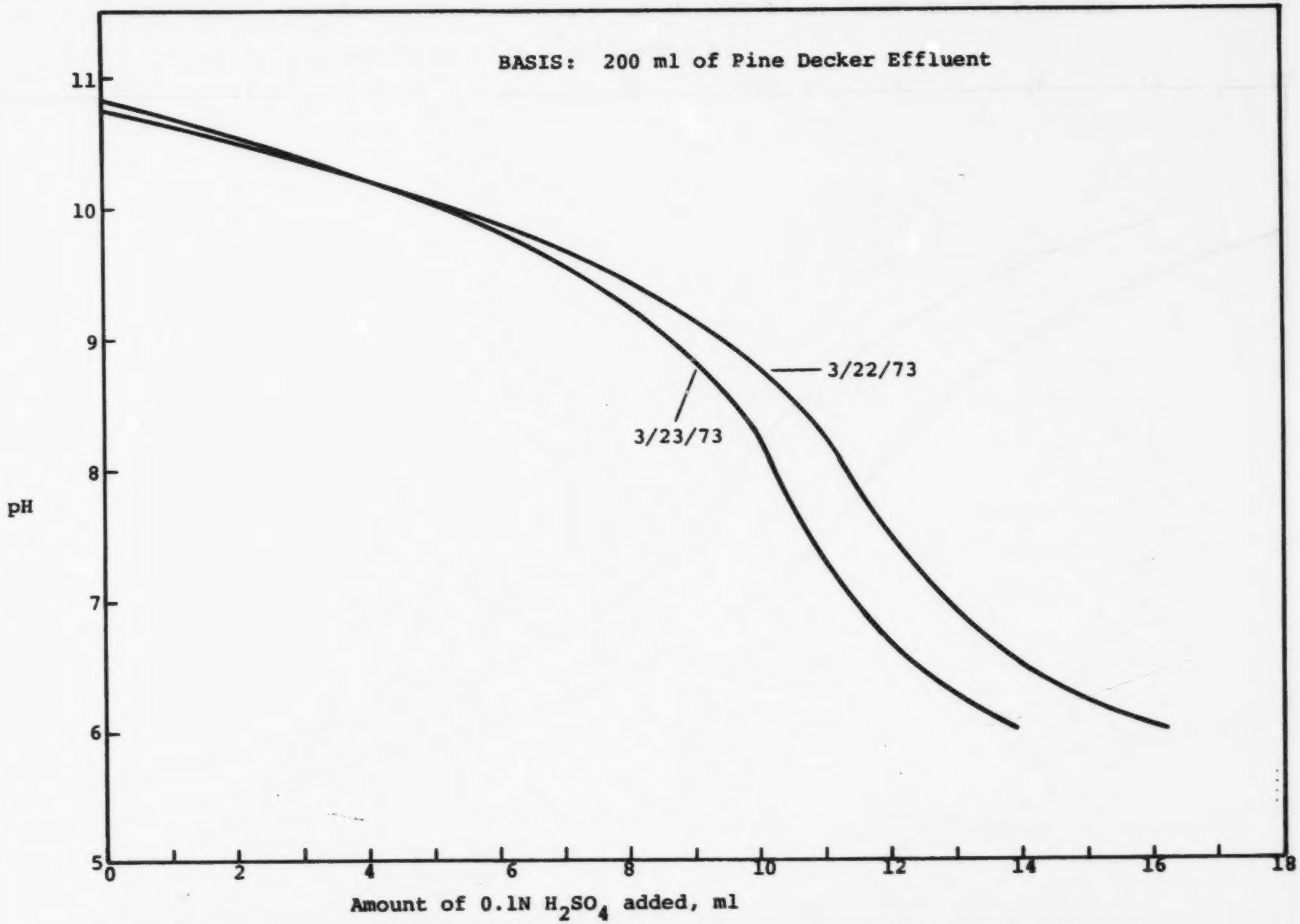


FIGURE 24. SULFURIC ACID REQUIREMENT TO NEUTRALIZE PINE DECKER EFFLUENT

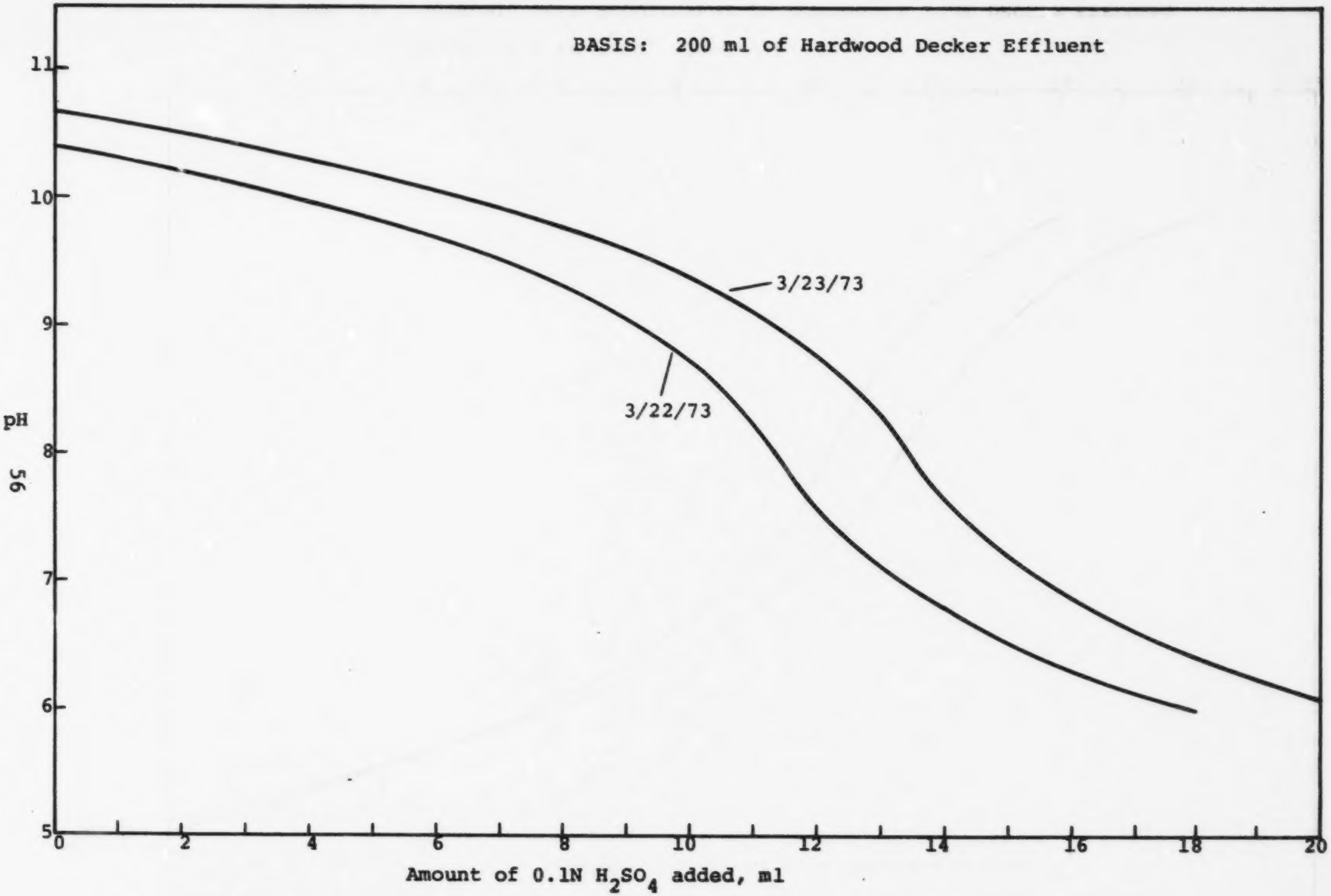


FIGURE 25. SULFURIC ACID REQUIREMENT TO NEUTRALIZE HARDWOOD DECKER EFFLUENT

effluent, and hardwood decker effluent respectively. About 4 lbs of 100% sulfuric acid are required to neutralize 1000 gals. of any one of the three effluents.

The major effects on feed characteristics of pH adjustment were:

- --a reduction in feed color;
- --an increase in the total solids of the feed by about 400-500 ppm, as a result of the addition of sulfate ion (from H_2SO_4); and
- --some change in the colloidal nature of the feed as evidenced by a change in suspended solids content.

Figure 26 shows the effect of pH adjustment on color for a sample of pine caustic extraction filtrate. The color, measured by sample absorbance at 625 mμ, gradually dropped with decreasing pH until at pH 2 the sample became turbid due to heavy flocculation of organics.

It was observed during several experiments with pine caustic extraction filtrate that the suspended solids level increased with reduced pH. Typical data are given below.

SUSPENDED SOLIDS OF PINE CAUSTIC EXTRACTION FILTRATE AS A FUNCTION OF pH

pH Adjusted With H_2SO_4	Suspended Solids, ppm
8	43
7	39
6	58
5	113
2	turbid (not measured)

This behavior of pine caustic extraction filtrate is further discussed in Section V.E : Important Factors Controlling Membrane Flux.

3. Prefiltration

In a previous laboratory test program, the necessity of feed pretreatment for particulate removal to obtain satisfactory ultrafiltration rates was apparent. The

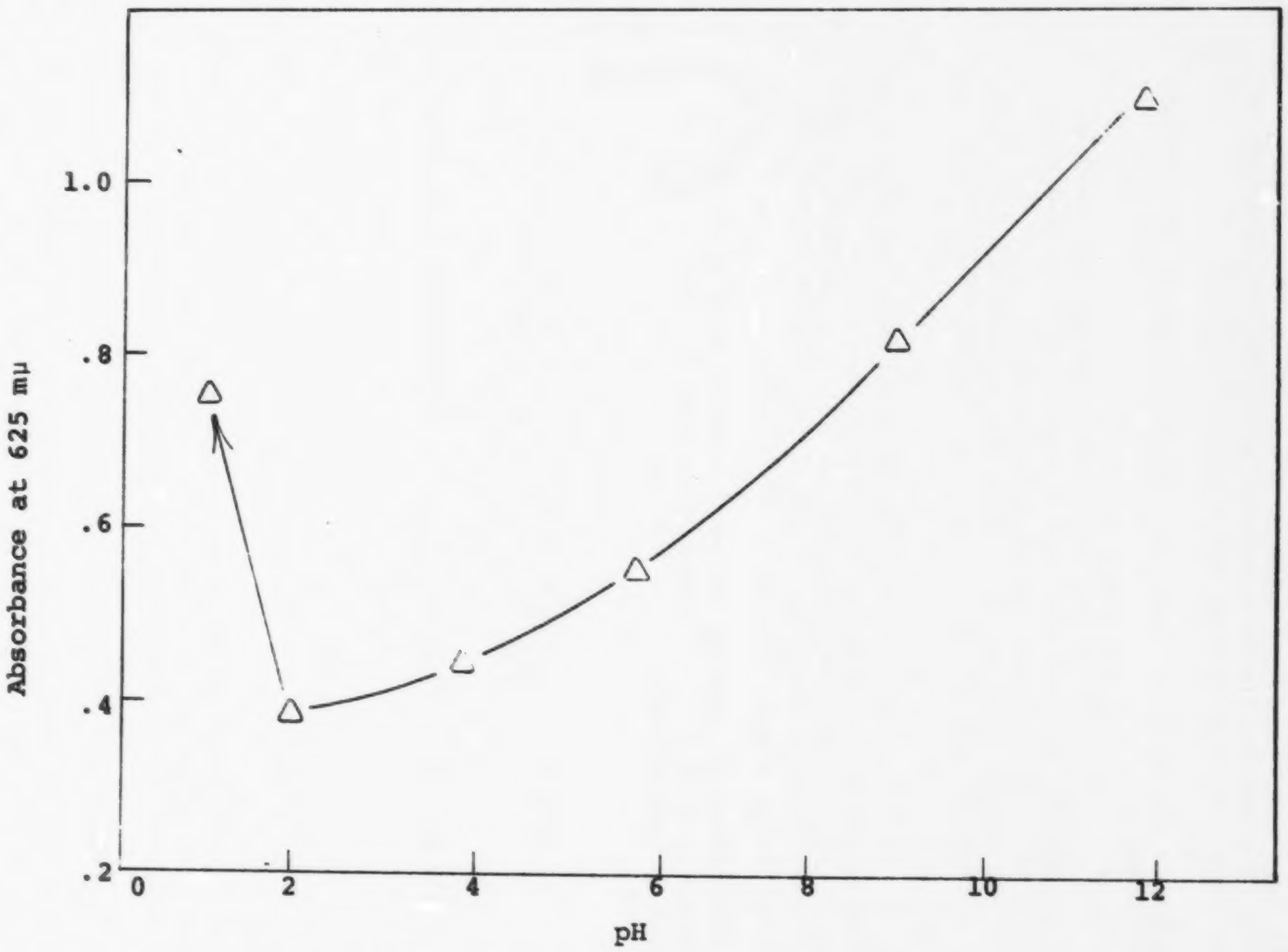


FIGURE 26. EFFECT OF pH ON COLOR OF PINE CAUSTIC EXTRACTION FILTRATE

exact type of filtration and filter equipment could not be determined under laboratory conditions with "aged" feeds. Hence, a substantial amount of time was devoted during this pilot plant program to investigate filters and their operating conditions in order to obtain satisfactory removal of suspended solids. The following filters and filter combinations were examined in the pilot plant program:

8/14/72-9/27/72	Broughton nominal 10μ mesh filter;
9/27/72-10/5/72	1) Bauer Hydrasieve (removal of fibers and gross suspended solids; 2) Hydromation depth filter (1-5μ solids removal); and 3) Cuno polishing filters (1μ cartridges;
10/17/72-2/1/73	1) Shriver filter press with wood flour as precoat material and filter aid (body feed, between 150 and 300 ppm); and 2) Cuno polishing filters (1μ cartridges;
2/2/73-3/1/73	1) Sparkler pressure leaf filter with wood flour as the precoat material and filter aid (body feed, between 150 and 300 ppm); and 2) Sparkler Velmac 5μ disc filter

Four different main filters were used: the Broughton mesh filter, the Hydromation depth filter, the Shriver filter press, and the Sparkler leaf filter. The installation of each was carried out to improve pilot plant performance. The Hydromation depth filter used a packed bed of granular PVC as the filtration medium. The Shriver and Sparkler filters were precoat filters and operated with filter aid added to body the feed. For most of the experimental program wood flour was used as the body feed; however, some tests were performed with other filter aids, and the results are discussed below.

a. Operational Experience

Immediately after pilot plant startup, it became apparent that the Broughton filter was not providing adequate suspended solids removal. Specifically, the membrane cartridges were plugging rapidly with suspended solids, as evidenced by a rapid increase in pressure drop across the cartridges and a rapid decrease in ultrafiltration rate. At that time a sample of the suspended solids in the pine caustic extraction filtrate was analyzed for particle size distribution. The results of this analysis are contained in Table 5. It is apparent that about 50% of the suspended solids are below 10μ, and is not surprising that the nominal 10μ Broughton filters proved ineffective for particulate removal. No actual data were obtained on the removal efficiency of suspended solids by the Broughton filter, but it was probably 50% or less.

Samples of the pine caustic extraction filtrate were submitted to filter vendors to obtain recommendations for prefiltration. Vendor tests showed that surface filtration resulted in rapid filter blinding, and that depth filtration was required. As an initial approach, a Hydromation depth filter was installed. Excellent removal of suspended solids was obtained, as determined both by the reduction in suspended solids level and more stable ultrafiltration rates in the pilot plant (data discussed in Section V.C.). Unfortunately, the filter size was inadequate for operation of the entire pilot plant on a continuous basis. The vendor was unable to deliver a larger filtration unit within the schedule and budget constraints of the program, and it was decided to switch to a precoat filter.

An existing Shriver filter press (75 ft^2 area) was available within the mill, and was installed in the pilot plant. Based on a recommendation by the Sparkler Manufacturing Co., Wilner wood flour # 139 was chosen as the precoat and body feed material. Substantially improved performance of the pilot plant was obtained with the Shriver filter press compared to the Broughton basket filters. Pilot plant operation continued for about 3-1/2 months using the Shriver filter. However, it was realized in December that the filter was operating at an unrealistically low filtration rate ($\sim$ 0.1 gpm/ft^2) and it was decided to install another filter which could treat the feed at more typical filtration rates

TABLE 5

PARTICLE SIZE DISTRIBUTION OF SUSPENDED SOLIDS IN PINE CAUSTIC EXTRACTION FILTRATE (UNNEUTRALIZED)

Particle Size (microns)	Weight, %	Cumulative Weight, %
>149	9.9	99.99
88-149	2.8	90.09
44-88	3.9	87.29
40-44	1.03	83.39
32-40	1.88	82.36
20-32	13.76	80.48
10-20	17.68	66.72
5-10	19.84	49.04
2.5-5	20.94	29.2
<2.5	8.26	8.26

($\sim$ 1 gpm/ft^2). Furthermore, it was suspected that the Shriver press did not provide the quality of filtration normally obtained with a precoat filter. This conclusion was based on often erratic performance of the ultrafiltration unit. The following considerations may explain the less than adequate performance of the Shriver filter:

--it was an old filter and possibly contained defects;

--the flow distribution between the different plates and frames (24 plates) was poor and the precoat thickness was probably not uniform. Furthermore it was not possible to form the precoat under high flow conditions which would have produced a more uniform precoat;

--the filter press was operated at high pressure drop, between 30 to 60 psi, and this could have resulted in the "extrusion" of solids into the filtrate;

--operational problems may have allowed the precoat to fall off when switching valves to change from precoat application to treating neutralized feed.

For these reasons a Sparkler leaf filter was ordered and installed at the beginning of February, 1973. The Sparkler filter provided excellent suspended solids removal and filtration rates. It was possible to maintain filtration rates of approximately 1 gpm/ft^2 for periods of up to 24 hrs, depending on the feed type and characteristics. (Note, filtration characteristics of the caustic extraction filtrate and decker effluents differed, as discussed below).

Table 6 contains a summary of the performance data of the main filters in processing pine caustic extraction filtrate. The data for removal of suspended solids are based on a suspended solids analysis by filtration on 0.45μ Millipore filters. As described before, this method provided only approximate values for suspended solids levels. However, it is possible to recognize major trends in filter performance. Based on the suspended solids removal data in Table 6, it was judged that the Sparkler and Hydromation filters gave the best performance; and performance of the Shriver filter press was good at times, but erratic. The filtration rates of both the Sparkler and Hydromation filters are judged to be in the range suitable for commercial application.

TABLE 6

PERFORMANCE - MAIN FILTERS

Filter	Filter Area sq ft	Filter Medium	Max/Min Flows During Operation, gpm	Max/Min Filtration Rates During Operation, gpm/sq ft	Min/Max Duration Filtration Cycle, hrs	Filtration Efficiency as indicated by Subsequent Ultra-filtration Rates	Removal of Suspended Solids % Removed
Broughton	5.6	10μ mesh	8-3	1.5-0.5		inadequate	
Hydromation	1	granular PVC	8-3	8-3	0.1-1.0	acceptable	50-80
Shriver	75	wood flour	10-3	0.13-0.04	4-20	sometimes acceptable	20-80
Sparkler	15.3	wood flour	25-5	1.6-0.3	4-16	acceptable	50-80

Perhaps the most relevant information on suspended solids removal by the different filters is obtainable from the ultrafiltration rates of the pilot plant. In general, the ultrafiltration rate data show that the Sparkler pressure leaf filter and the Hydromation depth filter were equally effective in removal of suspended solids. At times the Shriver filter press performed adequately.

b. Performance of Other Filters

The performance characteristics of the Bauer Hydrasieve, the Cuno cartridges, and the Sparkler Velmac disc filter were not closely monitored because of their relatively limited influence on the performance of the ultrafiltration unit.

The Bauer Hydrasieve adequately removed fibers and gross suspended solids from the raw feed, which prevented clogging of valves and pumps used before the main filter. However, the performance of the Bauer Hydrasieve was not a critical part of the filtration operation from the point of view of ultrafiltration performance.

The Cuno cartridge filters were installed just before the ultrafiltration unit for final polishing of the feed. These filters also served to protect the ultrafiltration unit in case of gross failure of the main filter. The Cuno filters were primarily used in combination with the Shriver filter press. Some additional suspended solids removal was achieved by the Cuno filters and it was necessary to change the disposable cartridge elements every 5-10 hrs of operation (1000-3000 gals.). It cannot be ascertained whether the solids removed by the Cuno filters are particulates which would pass through any precoat filter, or are suspended solids which passed through the Shriver filter due to below-par performance.

In the final phase of program, the Cuno filters were replaced by a Sparkler Velmac disc filter because of the larger filter area and the capability of cleaning the filter discs. In combination with the Sparkler leaf filter, the Velmac filter lasted for about 60 hrs (20,000 gal.) when it was used for the first time. Most of the solids removed by the Velmac filter could be removed by washing. However, some reduction in filter capacity was observed in subsequent runs due to an inability to completely regenerate the filter medium. The ultimate useful life of the Velmac filter has not yet been established, but it is anticipated that in operation of a full-scale plant, life will be economically acceptable.

c. Tests with Different Filter Aids

Two sets of experiments were performed. One with the Shriver filter press, and another with the Sparkler leaf filter.

The data from the Shriver filter tests were obtained under conditions of low filtration rates (~0.1 gpm/ft^2), and can only be used as a guide in evaluating performance characteristics of different filter aids. In these tests, five different filter aid materials were used: # 139 wood flour, (Wilner Wood Products, Norway, Maine), Dicalite 436 (Grefco diatomaceous earth), Celite 545 (Johns-Manville diatomaceous earth), Hyflo Super Cel (Johns-Manville), and Perlite 400F (Chemrock Corp.). In the tests a precoat of about 6 to 10 lbs was applied to the filter, and the filter aid was added as body feed at a level of approximately 150 ppm. In these tests, no observable difference in removal efficiency of suspended solids was noted. However, a substantially longer filtration cycle was obtained with the wood flour than with the other filter aids. This suggests that the filter cake formed with wood flour is less susceptible to plugging or blinding by the suspended solids-filter aid suspension.

In the tests with the Sparkler filter, three different types of filter aid were examined: wood flour, diatomaceous earth (two grades), and a Perlite. The tests were performed with two different effluents: pine caustic extraction filtrate and hardwood decker effluent. In these tests, a 3-lb precoat of each of the filter aid materials was applied to the 15.3 ft^2 Sparkler filter. After the precoat was applied, wood flour was used as the bodying material, and added at a level of approximately 150 ppm.

The effluent filtered was always fresh, and was withdrawn directly from the 500 gal. feed tank. Operation was at a standard filter pressure drop of 20 psi (except for the one run with Hyflo Super Cel since this is a looser type of filter aid and could compress at a 20 psi pressure differential.

A summary of the test results is presented in Table 7.

The data in Table 7 show that initial filtration rates for all of the precoat materials were excellent, in the range of 1.4 to 1.8 gpm/ft . In the tests with the pine caustic extraction filtrate, wood flour and the Dicalites maintained their original filtration rates over the full 21 minute test period. In the tests with the hardwood decker effluent, which contained a much higher level of suspended solids, an appreciable falloff in filtration rate was observed, as expected.

The Hyflo Super Cel is a much coarser filter aid, with a greater porosity and higher initial filtration rate. However, the filtration rate dropped off sharply with both pine caustic extraction filtrate and hardwood decker effluent, indicating that a fairly dense filter aid is to be preferred as a precoat medium if long filtration cycles are to be achieved.

Removal of suspended solids in all tests was about the same, approximately 50 to 75%.

On the basis of the tests with both the Shriver and the Sparkler filters, it was concluded that the Wilner # 139 wood flour was the preferred filter aid. In addition, wood flour has the advantages that it is inexpensive and can be easily disposed of by incineration with the concentrate from the ultrafiltration unit. For these reasons, wood flour was used for the experimental program.

4. Feed Characteristics

The detailed experimental data on feed characteristics for caustic extraction filtrate, pine wood decker effluent and hardwood decker effluent, respectively, are presented in summarized form in Table 8.

TABLE 7

TESTS WITH DIFFERENT FILTER AIDS ON SPARKLER 15.3 sq ft LEAF FILTER

Filter Aid	Type of Filter Aid	Effluent	Pressure Drop psi	Precoat, lbs	Filtration rate gpm/ft^2		Suspended Solids in/out ppm
					initial	final (21 min)	
Wilner #139	wood flour	Pine caustic extract	20	3	1.4	1.4	132/47
Dicalite 436	diatomaceous earth	"	20	3	1.7	1.5	20/10
Dicalite 436	"	"	20	3	1.5	1.6	75/18
Dicalite 476	"	"	20	3	1.6	1.6	44/20
Hyflo Super Cel	Perlite	"	10	3	1.6	0.85	50/19
Wilner #139	wood flour	Hardwood decker effluent	20	3	1.4	0.9	300/160
Dicalite 476	diatomaceous earth	"	20	3	1.8	1.2	260/180
Hyflo Super Cel	Perlite	"	20	3	1.5	0.2	280/100

The range of compositions shown in Table 8 are based on analyses of all samples collected throughout the pilot plant program. The detailed data presented in Figures 27 to 33 show that the feed composition varied widely over relatively short periods of time, often within the same day. Sharp variations in feed composition were noticed particularly when start up or shutdown operations took place in the mill. This alone suggests that improved effluent flow control could reduce the mill washwater volume. Presumably operation at high solids-high color loadings did not impair pulp or paper quality. It should be possible to maintain these loadings near the highest tolerable level, thus reducing water consumption.

a. Suspended Solids

Table 8 and Figures 27 to 29 give the ranges of suspended solids that were determined for the three effluents, both untreated and pretreated. As discussed in the section on Analytical Methods, the suspended solids data are only approximate, and this accounts, in part, for the scatter in the data.

The suspended solids level of the hardwood decker effluents, during the limited time when measurements were made, was found to be higher than those for the other two effluents.

b. Total Solids

Figures 30 and 31 show the total dissolved solids contents of pine caustic extraction filtrate, pine decker effluent, and hardwood decker effluent, respectively. Pine caustic extraction filtrate had a much greater amount of total solids (avg. about 7000 ppm) compared to the decker effluents which averaged about 2500-3000 ppm solids.

Feed neutralization and prefiltration introduced about 400-500 ppm of solids into the three effluents, due to the addition of sulfate ion.

TABLE 8

SUMMARY OF FEED CHARACTERISTICS

	Pine Caustic Extraction Filtrate		Pine Decker Effluent		Hardwood Decker Effluent	
	Range	Average	Range	Average	Range	Average
1. pH						
Untreated Feed	11.5-12	-	10.7-10.9	-	10.0-10.6	-
Treated Feed*	6.0-6.5	-	6.0-6.5	-	6.0-6.5	-
2. Color, ppm, Cobalt units						
Untreated Feed	12,000-45,000	28,000	4,000-9,300	6,000	8,000-22,000	11,000
Treated Feed*	10,000-27,000	19,000	3,000-7,000	4,000	6,000 13,000	8,000
3. Total solids, ppm						
Untreated Feed	4,400-11,400	7,000	1,700-8,200	2,400	1,200-4,600	3,000
Treated Feed*	**	**	**	**	**	**
4. Suspended Feed, ppm						
Untreated Feed	50-200	80	50-150	80	100-350	200
Treated Feed*	10-90	30	20-40	(limited data)	50-180	(limited data)

* pH of feed adjusted to 6.5-6.9, followed by filtration through a depth or precoat filter

** total solids in neutralized, filtered feed were increased over untreated feed by about 400-500 ppm due to sulfate addition (H_2SO_4)

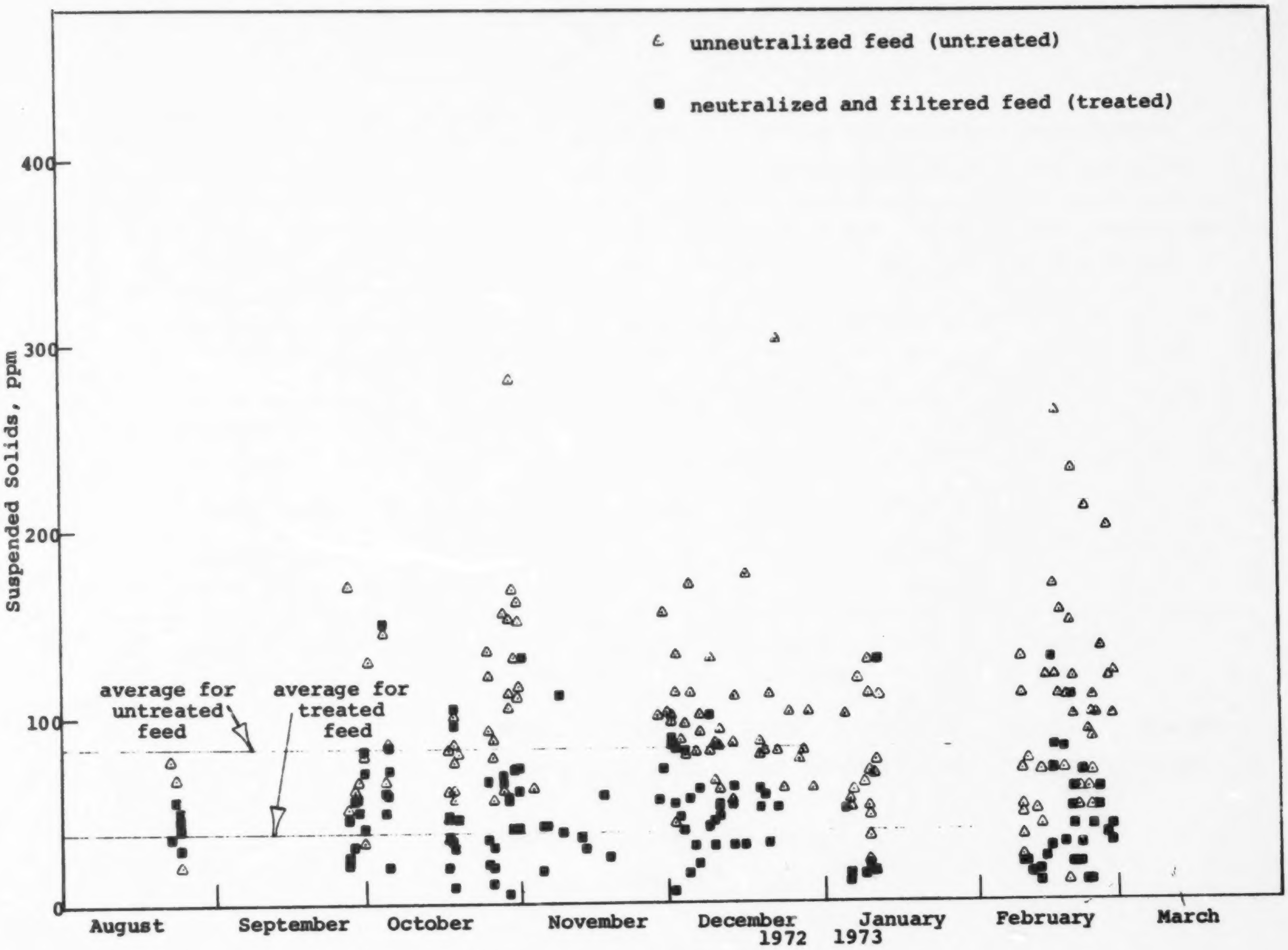


FIGURE 27. SUSPENDED SOLIDS OF CAUSTIC EXTRACTION FILTRATE

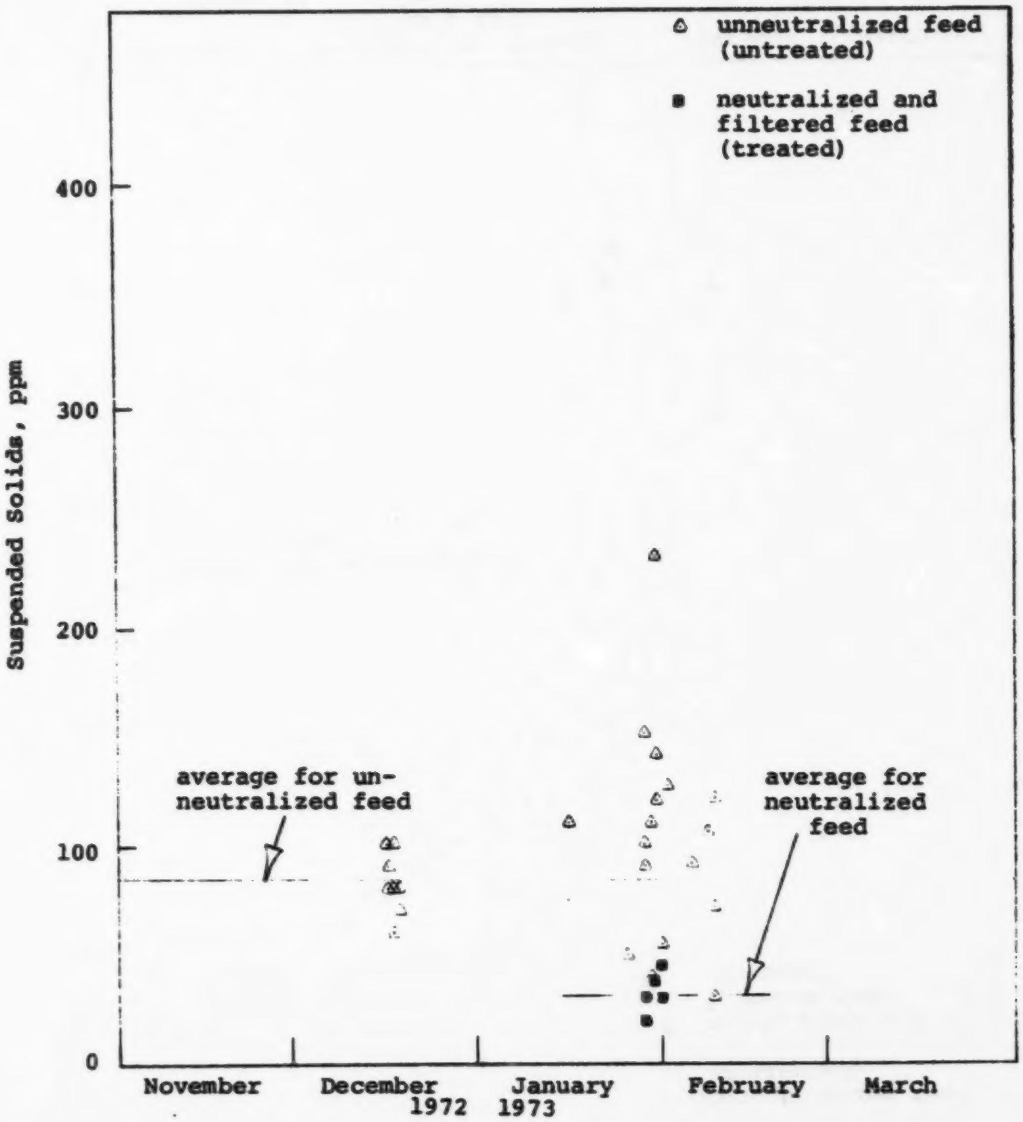


FIGURE 28. SUSPENDED SOLIDS OF PINE DECKER EFFLUENT

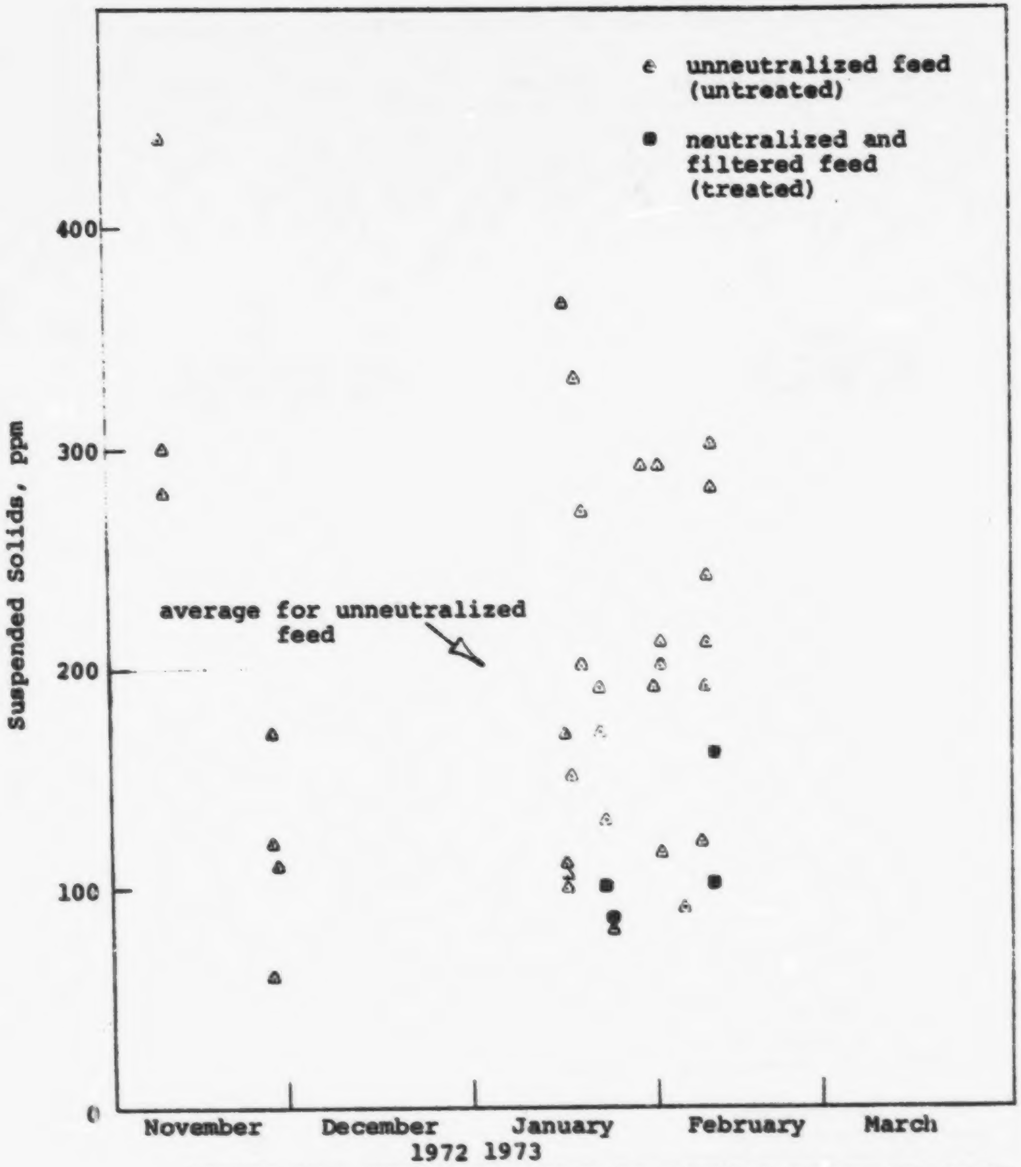


FIGURE 29. SUSPENDED SOLIDS OF HARDWOOD DECKER EFFLUENT

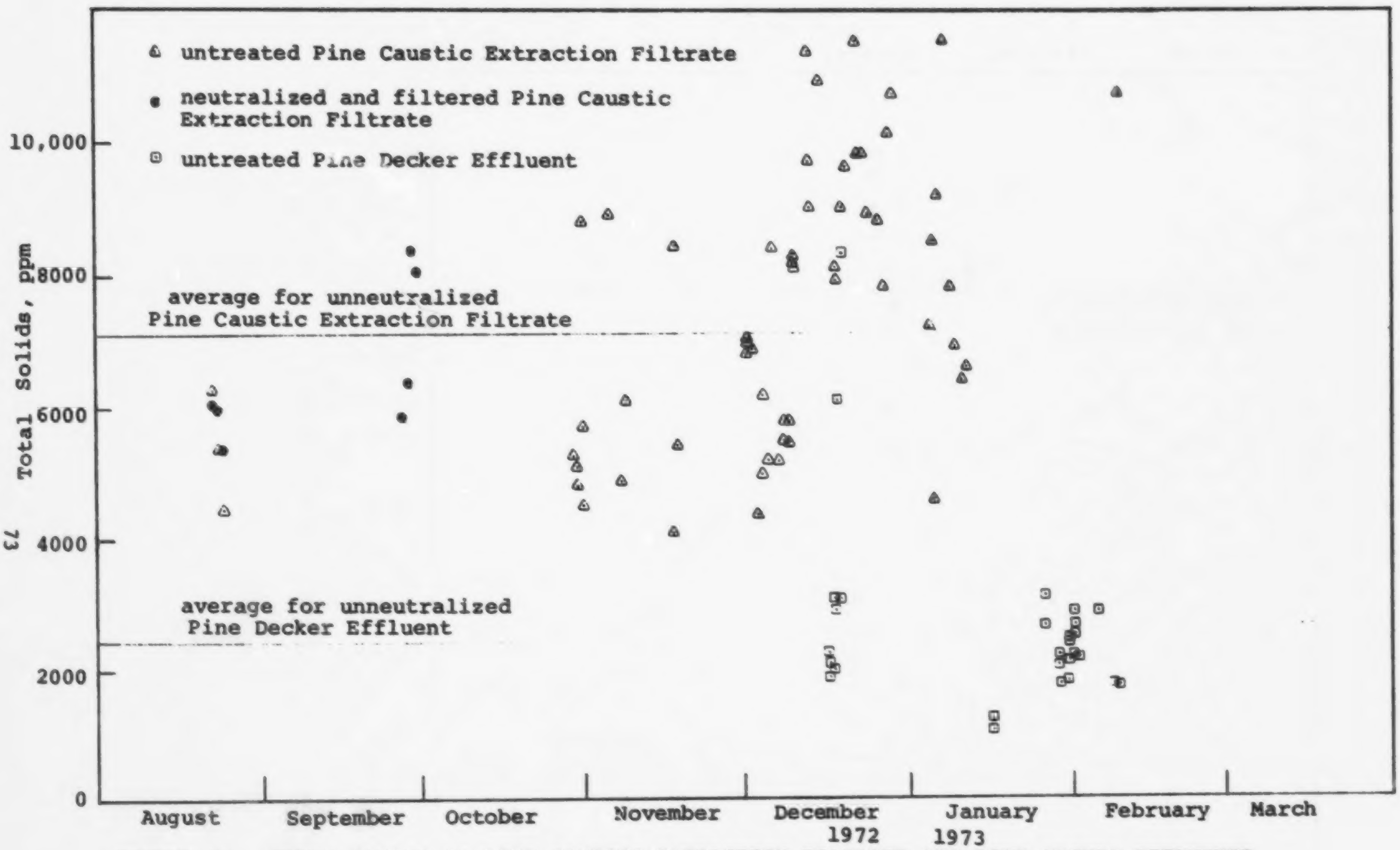


FIGURE 30. TOTAL SOLIDS OF PINE CAUSTIC EXTRACTION FILTRATE AND PINE DECKER EFFLUENTS

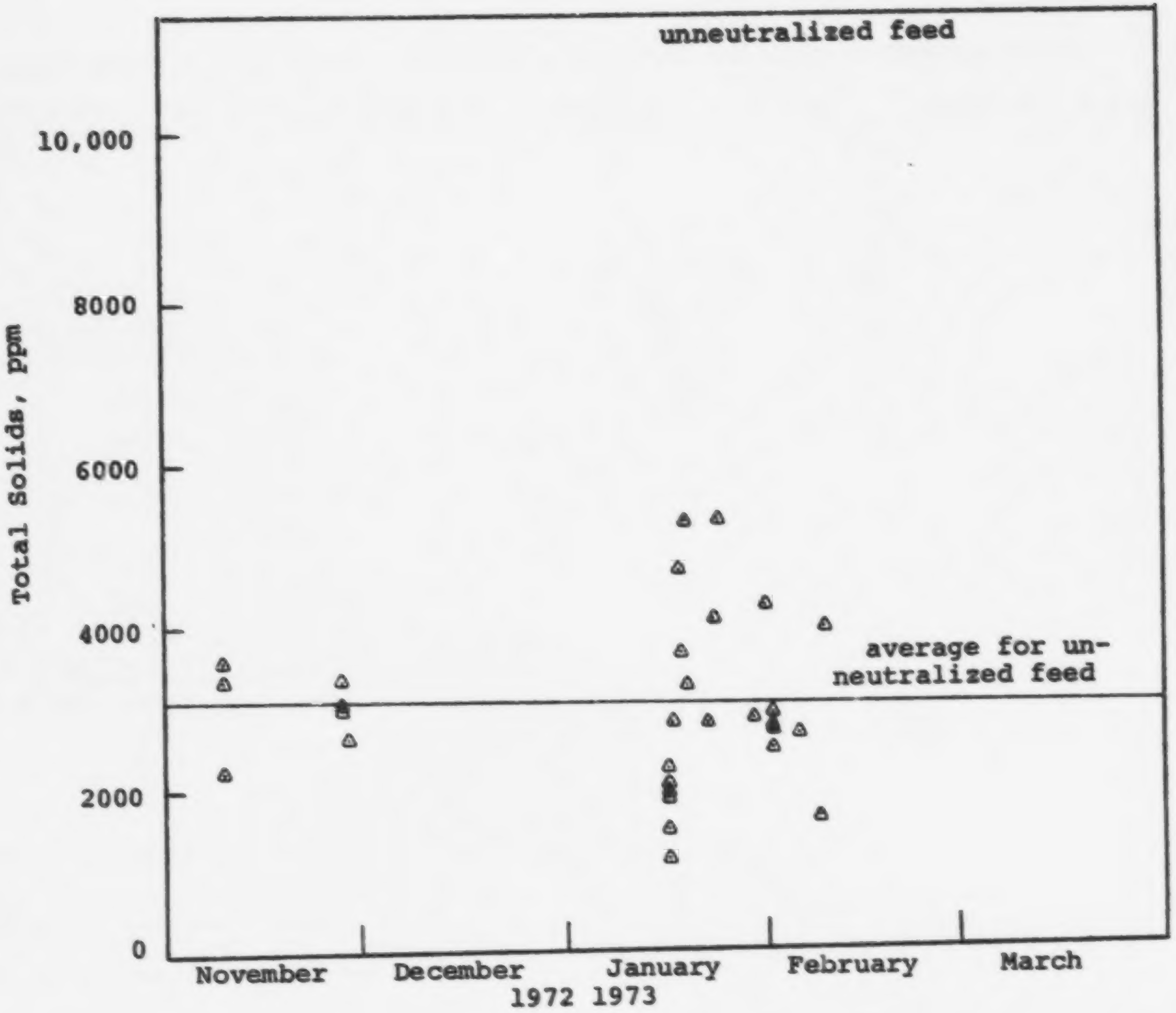


FIGURE 31. TOTAL SOLIDS OF HARDWOOD DECKER EFFLUENT

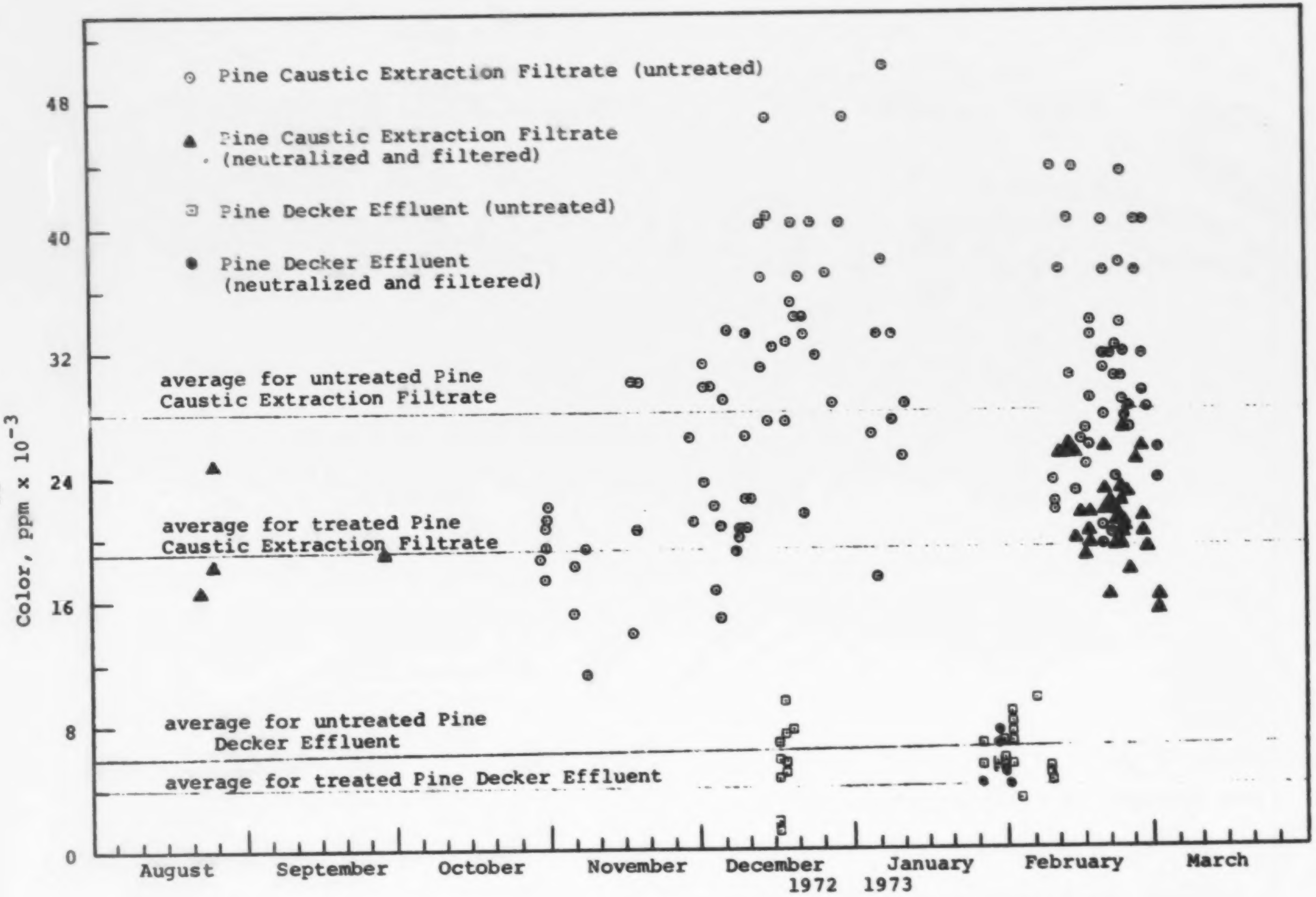


FIGURE 32. COLOR OF PINE CAUSTIC EXTRACTION FILTRATE AND PINE DECKER EFFLUENT

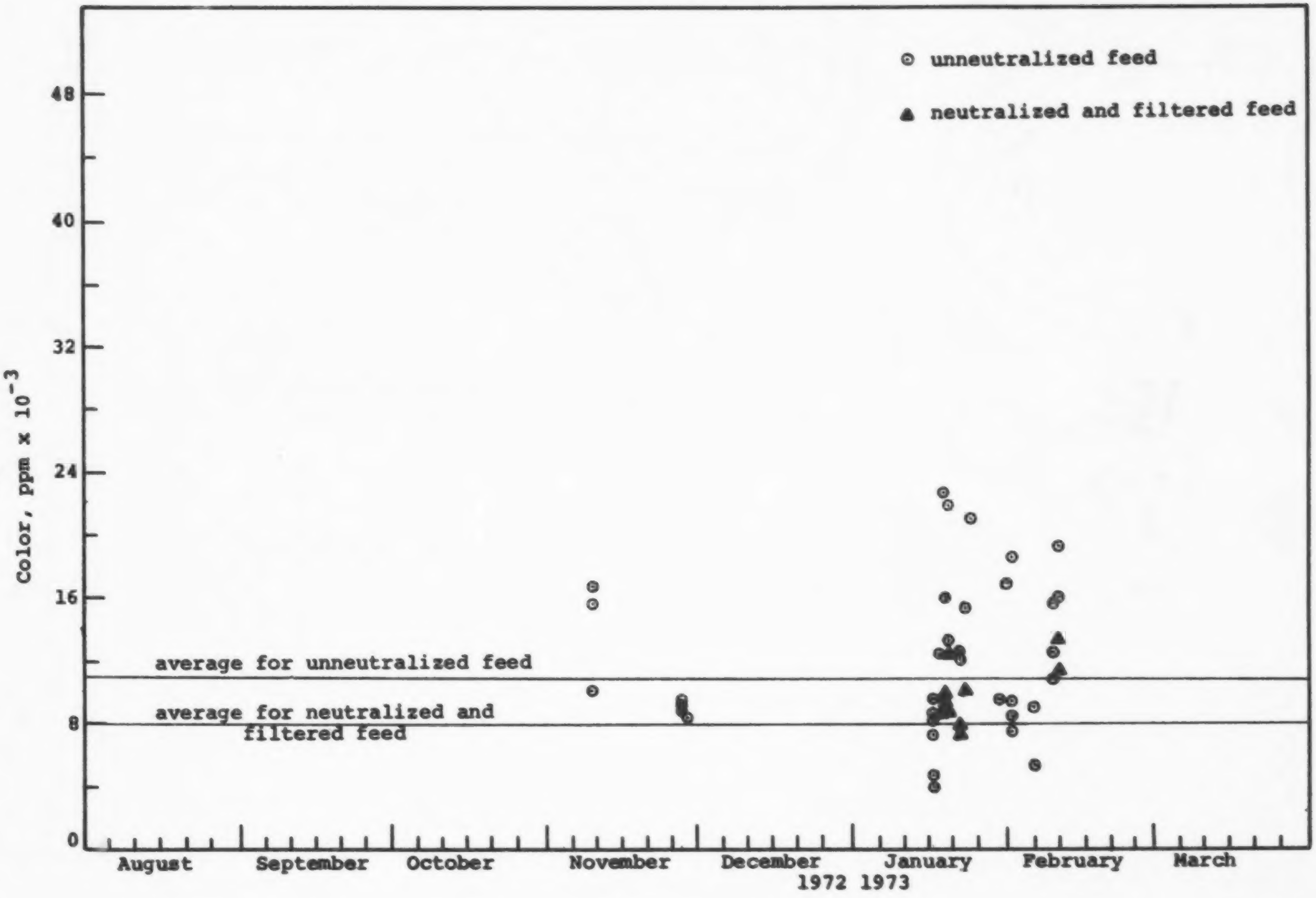


FIGURE 33. COLOR OF HARDWOOD DECKER EFFLUENT

c. Color

Figures 32 and 33 show the color contents of the three effluents. It is seen from these figures that the pine caustic extraction filtrate was much more highly colored (avg 28,000 ppm), than the other two effluents. Pine decker effluent had the least color, averaging about 6000 ppm.

Figure 34 shows the effect of feed pretreatment (pH adjustment to 6.5 to 6.9 and precoat filtration) on the "field" color content of a series of samples of pine caustic extraction filtrate. Feed pretreatment removed about 30-35% of the color from the raw feed; the major part was due to pH adjustment.

d. Other Solutes

Table 9 gives additional analytical data for a few feed samples of the three effluents.

In summary, the following conclusions can be drawn about the characteristics of the three effluents:

- --Pine caustic extraction filtrate has higher levels of total dissolved solids, ash and color than either of the decker effluents;
- --Although the two decker effluents have similar levels of total dissolved solids, pinewood decker effluent has less color;
- --Hardwood decker effluent has the highest suspended solids level; pine caustic extraction filtrate and pine decker effluent levels are about the same;
- --Pine caustic extraction filtrate contains much more total and ionic chloride than the decker effluents;
- --Decker effluents contain a substantial amount of sulfate ion, originating in the pulp digester;
- --The feed compositions of all three effluents vary widely within a relatively short period of time;
- --Pretreated effluents have about 30-35% less color than the untreated (raw) effluents;
- --pH adjustment of the effluents introduces about 400-500 ppm sulfate ion;
- --A major fraction of the suspended solids of the three effluents is smaller than 10µ, but is reasonably effectively removed by either depth

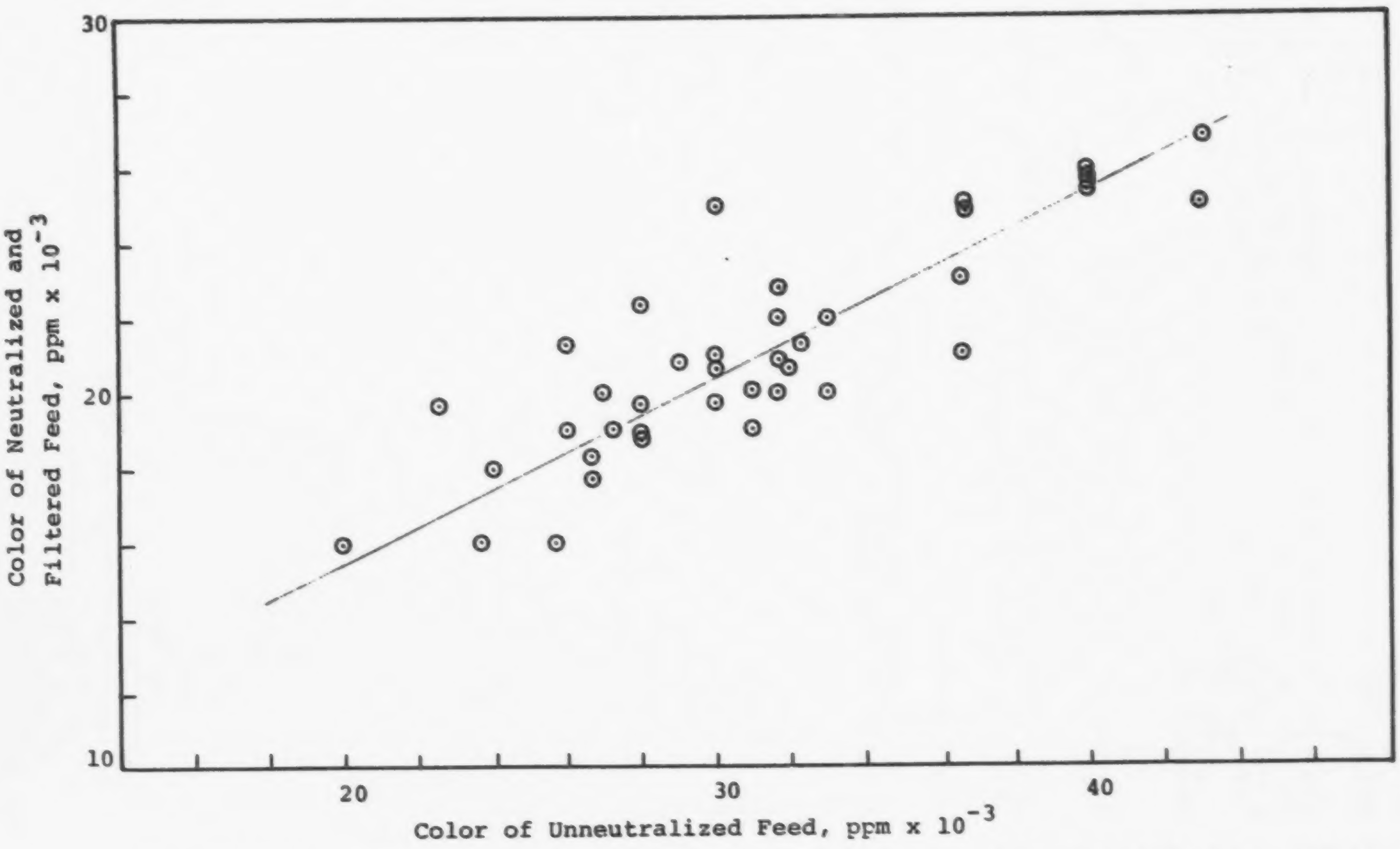


FIGURE 34. EFFECT OF FEED PRETREATMENT ON COLOR OF PINE CAUSTIC EXTRACTION FILTRATE

TABLE 9

WASTE CHARACTERISTICS AT THE NORTH CAROLINA MILL - DETAILED ANALYSES

Components	Untreated Pine Caustic Extraction Filtrate			Untreated Pinewood Decker Effluent sampled on 1-3-73			Untreated Hardwood Decker Effluent sampled on 2-1-73		
	10-13-69	10-16-70	2-10-73	3 pm	5 pm	8 pm	1 pm	3 pm	5 pm
pH	11.3	11.5	11.7	10.7	10.7	10.7	10.0	10.5	10.7
Color, ppm		14,000	28,000	7,700	7,300	8,300	18,300	8,300	7,700
S.S., ppm			80	230	70	140	400	114	214
T.S., ppm		6,800	7,000	2,244	2,284	2,468	3,848	2,520	2,244
Ash, ppm		3,800		1,010	784	908	976	848	1,464
Na^+, ppm	1,180	2,000	1,200	480	425	516	58	480	441
Ca^{++}, ppm	78	5	60	20	16	4	16	4	12
Cl^-, ppm	1,010	1,600	1,275	14	14	14	30	6	35
SO_4^{--}, ppm				260	190	240	350	200	200
Fe^{+++}, ppm		1.6		1.4	1.2	0.8	2	1.2	0.1

filtration or precoat filtration.

B. REJECTION DATA AND EVALUATION

Comprehensive data were collected on detailed color and total solids rejections data for pine caustic extraction filtrate, pine decker effluent and hardwood decker effluent, respectively. Specifically, the following information was included:

a. Color of unneutralized feeds; neutralized and filtered feeds, final concentrates and mixed permeates from the ultrafiltration unit;

b. Total solids of unneutralized feeds, final concentrates and mixed permeates from the ultrafiltration unit;

c. Concentration ratios, defined as the ratio of feed flow to the ultrafiltration unit to concentrate flow from the ultrafiltration unit;

d. Percent color removal, defined as

$$\frac{\text{color of unneutralized feed-color of mixed permeate}}{\text{color of unneutralized feed}} \text{ x } 100;$$

and

e. Color removal efficiency of the ultrafiltration unit by stage for treatment of pine caustic extraction filtrate.

For presentation here these data have been condensed and displayed in Figure 35, which summarizes the separation efficiency of the pilot plant during the operating period. For all three effluents, the concentration ratio achieved, the total solids level in the concentrate, and the percent color removal are shown.

1. Color Rejection

As seen in Figure 35 excellent color removal was obtained. For example, for about eighty-fold concentration, color removal was approximately 90% for the pine caustic extraction filtrate and 95% for both decker effluents. The higher color removal efficiency for decker effluents is hypothesized to be related to the molecular weights of

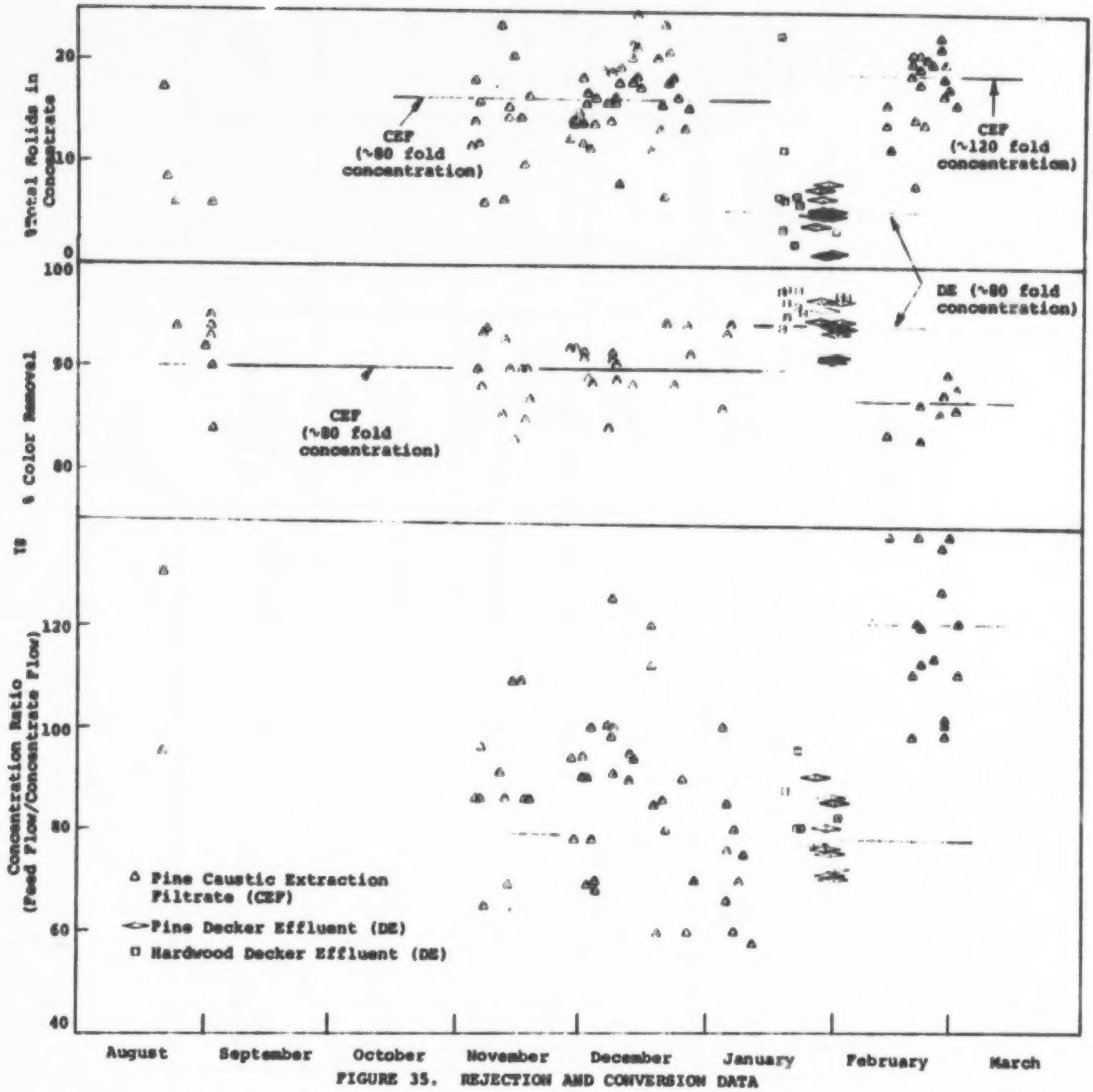


FIGURE 35. REJECTION AND CONVERSION DATA

Table 10

REJECTION

Effluent	Period	Color Concentration, ppm				Total Solids in Concentrate %	Concentration Ratio	Color Removal, %
		Raw Feed	Neutralized Filtered Feed Average	Permeate	Final Concentrate			
Pine Caustic Extraction Filtrate	11/5-11/8/72	14,000 18,000	11,000	1200-1500	500,000-600,000	12-16	80-90	92
	11/28-12/1/72	20,000 30,000	17,000	2000-3000	1,000,000-1,500,000	12-15	80-100	90
	2/22-2/27/72	30,000-40,000	23,000	4000-5600	1,600,000-2,500,000	18-22	100-130	86
Pine Decker Effluent	1/30-1/31/73	6,000-8,000	4,500	430-470	180,000-220,000	5	70-90	94
Hardwood Decker Effluent	1/18-1/23/73	12,000-16,000	9,000	600-900	420,000 500,000	6-7	80-90	95

the color bodies in the effluents. The presence of lower molecular weight lignaceous materials in the pine caustic extraction filtrate would be expected due to lignin fragmentation in the bleach plant chlorination stage.

The color removal was also dependent on the concentration ratio obtained: the greater the concentration ratio, the higher the color content of the mixed permeate. This is to be expected since permeate color increases with feed color, and at high conversion a greater fraction of the membrane area is exposed to highly colored feed.

This effect is further demonstrated by Table 10, which contains rejection data for selected periods when operation proceeded smoothly. Also shown is the higher color removal observed when processing decker effluents.

Appendix I contains some limited color rejection data for individual membrane stages when treating pine caustic extraction filtrate. Color rejection increased with feed concentration (progressing through the stages of the pilot plant). This is due to the fact that with increasing feed concentration a higher fraction of the feed color bodies are high molecular weight species since lower molecular weight solutes are preferentially removed in the earlier stages.

In addition, color rejection was clearly dependent on membrane type. The T.J. Engineering modules showed a wide variation in color rejection, between 87 and 96%, which is attributed in part to poor quality control in both membrane and cartridge manufacture, as well as occasional leaks. The color rejection of the four Gulf Environmental Systems modules was between 98-99.9%, and clearly superior to rejections for either the Eastman or T.J. Engineering modules. This was expected since the HT-00 membrane (used in the latter two modules) intrinsically had a higher "molecular weight cutoff" than the Gulf membranes.

Color rejection of the pilot plant was low on a few occasions due to mechanical failures of several T.J. Engineering cartridges: Especially troublesome was o-ring failures on the permeate collection tube (see comments column in Appendix D, and discussion in section V.G., Module Mechanical Failures).

2. Total Solids Rejection

Figure 35 also shows the effect of concentration ratio on total solids content of the final concentrate. At eighty-fold concentration of pine caustic extraction filtrate, the final concentrate contained about 15% total solids. The greater part of the solids were high molecular-weight organics since the ultrafiltration membranes used did not appreciably retain salts and low-molecular solutes. The detailed pilot plant data demonstrate that the total solids rejections of the membranes were between 5 and 30%.

3. Material Balances

Material balance data for color and total solids was collected and evaluated at least every operating shift. Both field color tests (at as-is pH) and the standard color tests (at pH 7.6) methods were used. Because of the pH sensitivity of the color values in the effluents, the standard test data was used for color balance calculations.

Tables 11 and 12 contain composition data of samples from tests with pine and hardwood decker effluents. These data also show that retention of non-complexed salts and other low molecular solutes (sodium ion, sulfate ion, ash and total solids) was real, but low. This can be seen best by comparison of permeate and concentrate assays. This was probably due to the use of the Gulf modules in Stages 4 and 5. These membranes had about 30% NaCl rejection.

Rejection of Fe^{+++} and Ca^{++} was very high, and this is attributed to complex formation of these ions with retained organics. The pH of concentrate samples was consistently lower than the filtered feed; and that for permeate samples was consistently higher. Evidently, the retained organics were weakly acidic.

The assays in Table 13 for pine caustic extraction filtrate samples also show similar behavior. Note values for the contents of trivalent metal oxides (mainly Fe^{+++}), Ca^{++}, $SO_4^{=}$, ash and chloride. Unfortunately, the samples were collected on different days and only qualitative conclusions can be drawn.

TABLE 11

COMPOSITION DATA: PINE DECKER EFFLUENT SAMPLES

Date Sampled 1-31-73 3:00 pm

Assays	Permeate	Concentrate (concentration ratio = 50)	Neutralized & Filtered Feed	Raw Feed
Total Solids	1,376 ppm	48,740 ppm	3,500 ppm	2,244 ppm
Ash	948 ppm	13,532 ppm	1,700 ppm	1,012 ppm
pH	7.0	5.9	6.5	9.5
Fe++	0.8 ppm	182 ppm	13.97 ppm	1.40 ppm
Na+	425 ppm	4,860 ppm	600 ppm	480 ppm
SO_4--	468 ppm	7,157 ppm	839 ppm	260 ppm
Ca++	0 ppm	448 ppm	16.05 ppm	20 ppm
Color	430 ppm	200,000 ppm		7,670 ppm

Date Sampled 1-31-73 5:00 pm

Assays	Permeate	Concentrate (concentration ratio = 50)	Neutralized & Filtered Feed	Raw Feed
Total Solids	1,296 ppm	49,312 ppm	3,624 ppm	2,284 ppm
Ash	868 ppm	13,652 ppm	1,684 ppm	784 ppm
pH	6.6	5.6	5.8	8.5
Fe++	0.6 ppm	110 ppm	6.20 ppm	1.2 ppm
Na+	325 ppm	4,620 ppm	575 ppm	425 ppm
SO_4--	470 ppm	7,280 ppm	900 ppm	190 ppm
Ca++	0 ppm	320 ppm	16.0 ppm	16.0 ppm
Color	430 ppm	200,000 ppm		7,330 ppm

Date Sampled 1-31-73 8:00 pm

Assays	Permeate	Concentrate (concentration ratio = 50)	Neutralized & Filtered Feed	Raw Feed
Total Solids	1,392 ppm	48,256 ppm	3,688 ppm	2,468 ppm
Ash	716 ppm	12,240 ppm	1,648 ppm	908 ppm
pH	6.5	5.3	5.8	9.2
Fe++	0.4 ppm	96 ppm	5.2 ppm	0.8 ppm
Na+	350 ppm	4,620 ppm	600 ppm	516 ppm
SO_4--	420 ppm	7,250 ppm	970 ppm	240 ppm
Ca++	0 ppm	280 ppn	8.0 ppm	4.0 ppm
Color	470 ppm	186,000 ppm		8,330 ppm

TABLE 12

COMPOSITION DATA: HARDWOOD DECKER EFFLUENT SAMPLES

Date Sampled 2-1-73 1:00 pm

Assays	Permeate	Concentrate (concentration ratio = 62)	Neutralized & Filtered Feed	Raw Feed
Total Solids	1,344 ppm	36,512 ppm	5,184 ppm	3,848 ppm
Ash	572 ppm	8,072 ppm	1,636 ppm	976 ppm
pH	6.8	5.8	6.0	10.5
Fe^{++}	1.2 ppm	134 ppm	5.2 ppm	2.0 ppm
Na^{+}	325 ppm	2,900 ppm	880 ppm	580 ppm
SO_4^{--}	200 ppm	4,000 ppm	350 ppm	350 ppm
Ca^{++}	0 ppm	96.0 ppm	24 ppm	16.0 ppm

Date Sample 2-1-73 3:00 pm

Assays	Permeate	Concentrate (concentration ratio = 60)	Neutralized & Filtered Feed	Raw Feed
Total Solids	2,316 ppm	59,652 ppm	6,296 ppm	2,520 ppm
Ash	1,192 ppm	12,896 ppm	2,280 ppm	848 ppm
pH	6.8	5.7	5.6	10.5
Fe^{++}	1.2 ppm	148 ppm	12 ppm	1.2 ppm
Na^{+}	558 ppm	4,500 ppm	920 ppm	480 ppm
SO_4^{--}	591 ppm	5,100 ppm	1,040 ppm	200 ppm
Ca^{++}	0 ppm	1,281 ppm	48 ppm	4.0 ppm
Color	730 ppm	400,000 ppm		15,000 ppm

Date Sampled 2-1-73 5:00 pm

Assays	Permeate	Concentrate (concentration ratio = 60)	Neutralized & Filtered Feed	Raw Feed
Total Solids	2,432 ppm	62,948 ppm	6,608 ppm	2,244 ppm
Ash	1,460 ppm	13,704 ppm	2,348 ppm	1,464 ppm
pH	6.9	5.7	5.8	10.7
Fe^{++}	1.0 ppm	125 ppm	7.0 ppm	0.1 ppm
Na^{+}	625 ppm	5,300 ppm	972 ppm	441 ppm
SO_4^{--}	660 ppm	5,950 ppm	1,600 ppm	220 ppm
Ca^{++}	0 ppm	481 ppm	80 ppm	12 ppm

TABLE 13

COMPOSITION DATA: PINE CAUSTIC EXTRACTION FILTRATE SAMPLES

ASSAY	PERMEATE	CONCENTRATE	
	sampled on 1-30-73	sampled on 2-13-73	sampled on 2-14-73
pH	7.4	6.7	7
Total Solids	4,560 ppm	142,850 ppm	200,000 ppm
Ash	2,950 ppm	27,920 ppm	35,000 ppm
Trivalent Metal Oxides	3.32 ppm	4,120 ppm	4,500 ppm
Na^+	1,200 ppm	10,400 ppm	12,000 ppm
SO_4^{--}	580 ppm	1,809 ppm	2,000 ppm
Ca^{++}	8 ppm	8,000 ppm	10,000 ppm
Cl^-	1,275 ppm	5,868 ppm	6,000 ppm
Specific Gravity	0.975	1.0635	1.0766

In summary, all the rejection data have confirmed the basic assumptions made at the beginning of the program. Color bodies and organics in the three effluents can be selectively concentrated by ultrafiltration. Permeates had low color contents, especially the decker effluents, but still contained the greater part of the total dissolved solids (ash) contained in the original effluents. High solids levels in the concentrate were achieved, at times over 20% in the pine caustic extraction filtrate tests.

C. ULTRAFILTRATION RATE DATA AND EVALUATION

The detailed ultrafiltration rate (flux) data have been condensed for presentation and discussion in this section.

The dependence of flux on time and operating conditions was complex. Operating time was important for both short-term operation, i.e. periods up to 48 hours, and long-term, i.e. months. Time effects can be attributed to four factors:

- --reversible membrane fouling/flow channel plugging (short term effect);
- --irreversible membrane fouling/flow channel plugging (long term effect);
- --membrane compaction (long term effect); and
- --membrane cartridge compression (long term effect).

During the pilot plant experiments, various changes were made in operating conditions and pilot plant equipment, including several changes of membrane cartridges. For this reason it is logical to discuss the operation of the pilot plant chronologically, and seven distinct periods are discussed below.

Before detailing performance of the pilot plant during these periods, however, flux data for the membrane system during the entire program will be presented. In Table 14 membrane flux by shell is presented, from the beginning of pilot plant operation through the end of the program. These fluxes were measured at the start of experiments; that is, after membrane cleaning. The efficiency of membrane cleaning was highly variable, and this explains in part some of the variation in day-to-day operation. These data have been included in Figures 36 to 42, which show the flux behavior at both the beginning and end of experiments. An evaluation of these data follows.

TABLE 14

MEMBRANE FLUX BY STAGE DURING PILOT PLANT PROGRAM

FEED*	Date	Cumulative Hours of Operation	Permeate Flux by Stage, gfd							Average flux, gfd
			1a	1b	1c	2	3	4	5	
PCEF	8-19-72	28	22.5	.75	.4	18	21	6	1.5	10.3
	8-21	32	24	16.5	6	18	24	7.5	3.0	14.4
	8-22	38	13.5	20.2	15	15	14.2	6.9	5.3	10.3
	8-25	50	12	12	7.5	7.5	15	7.5	4.4	10.3
	8-30	66	20.2	22.5	18	37.5	13.5	6	4.5	14.4
	8-31	80	10.5	8.2	7.5	9	10.5	9.3	2.7	8.2
	10-25	110	36	13.5	15	15	14.3	15	6.9	14.4
	10-28	135	30	7.5	12	15	12	15	9	14.4
	10-31	190	22.5		13.5		6.8	15	6.3	10.3
	11-1-72	200	34.5	34.5	33	34.5	30	30	15	29
	11-2	204	30	30	27	30	22.5	30	15	25.3
	11-2	220	28.5	28.5	28.5	28.5	24	28.5	15	25
	11-3	240	24	24	22.5	22.5	18	21	13.5	19.9
	11-6	280	18.9	18.9	21	22.5	21	15	4.5	16.6
	11-7	300	10.5	10.5	18.7	15	15	12.7	3.7	11.8
	11-10	336	12.9	12	22	18.7	20.2	22.5	6.6	15.8
	11-11	356	16.5	15	18.7	19.5	20.2	22.5	6.6	16.2

TABLE 14
(continued)

MEMBRANE FLUX BY STAGE DURING PILOT PLANT PROGRAM

FEED*	Date	Cumulative Hours of Operation	Permeate Flux by Stage, gfd							Average flux, gfd
			1a	1b	1c	2	3	4	5	
PCEF	11-12-72	376	13.5	12.7	17.2	16.5	15	15	8.9	13.6
	11-16		22.5	19.5	20.2	21	18.7	8.7	3.0	
	11-17	408	19.5	18	18	19.5	18	15	3.0	15
	11-18	430	19.5	15	18	15	14	14	2.4	13.4
	11-28	460	18	18	18	15	15	8.1	3.0	13
	11-29	476	10.5	10.5	9	15	12.7	6.0	2.4	8.8
	12-1	516	15	15	14	19.5	18	15	2.4	13.6
	12-2	530	15	15	21	18	15	15	4.5	14.2
	12-3	550	13.8	13	12	18.7	15	15	6.0	12.9
	12-5	585	17.4	15	15	18	15	17	7.3	14.3
	12-6	600	16.5	12.9	14.4	18	18	16.5	6	13.9
	12-7	615	13.8	11.7	11.9	15	18	12.4	2	11.5
	12-8	636	34.5	12	21	18	16.5	15	2	16.3
	12-9	644	33.8	12	18	15	18	15	2	15.6
	12-10	660	30	10.8	18	15	14	15	6.3	15
	12-11	680	34.5	12	19.5	15	16.5	13.8	5.4	16

TABLE 14
(continued)
MEMBRANE FLUX BY STAGE DURING PILOT PLANT PROGRAM

FEED*	Date	Cumulative Hours of Operation	Permeate Flux by Stage, gfd							Average flux, gfd
			1a	1b	1c	2	3	4	5	
PCEF	12-12-72	700	33	12	17.2	13.2	15	13.2	3.4	
	12-13	714	34.5	10.5	16.5	16.5	16.5		6.7	
	12-14	734	34.5	10.5	15	13.5	16.5		6.0	
	12-18	760	34.5	10.5	13.5	15	15		4.5	
	12-19	781	13.5	36	13.5	12	15		3.0	
	12-20	797			11.7	10.5	14.3		4.3	
	12-21	813		34.5	12	12	13.8	3.8	3.5	
	12-22	824		34.5	12.8	10.7	13.8	6	6	
	12-23	846		34.5		11.3	12.7	3.6	3	
	12-26	869		34.5		10.5	15	4.4	4.5	
	12-27	897		34.5		12	16.5	5.7	4.5	
	12-28	919		36		10.5	15	3.3	2.3	
	1-4-73	943		34.5		17.2	15	3.9	5	
	1-5	967		34.5		17.2	15	4.5	4.8	
	1-6	989		37.5		18.5	16.5	5	2.7	
	1-7	1010		34.5		18.5	16.5	3.3	1.5	
	1-8	1033		34.5		20.4	16.5	4.5	3.9	

TABLE 14
(continued)
MEMBRANE FLUX BY STAGE DURING PILOT PLANT PROGRAM

FEED*	Date	Cumulative Hours of Operation	Permeate Flux by Stage, gfd							Average flux, gfd
			1a	1b	1c	2	3	4	5	
PCEF	1-9-73	1056		31.5		18	17.2	4.5	3	
↓	1-10	1078		33		18.5	17.2	4.5	3.8	
HDE	1-18	1092		27.6		10.8	13.5	5.3	6	
↓	1-19	1098		22		9.6	13.5	5.3	6	
↓	1-22	1110		29.4		11.5	13	5.3	6	
↓	1-23	1120		26.4		9.9	11.4	4.2	3.8	
↓	1-24	1127		25.7		9.6	10.7	3.8	4.5	
PDE	1-26	1136		29.1		10.4	11.6	5.3	6	
↓	1-29	1146		29.1			12.9	4.5	5.3	
↓	1-30	1158		34.2			13	4.5	6	
↓	1-31	1172		27.3			11.1	4.5	4.5	
HDE	2-1-73	1184		26.9			12.4	5.3	4.5	
MDE	2-2	1196		24			10.5	4.5	4.5	
PCEF	2-12	1000		24.8			13.8	15		16
↓	2-13	1016		25.5	22.5		13.5	15	13.5	16.2
↓	2-19	1025	22.5	26.3	22.5		15	15	5.4	16.2
↓	2-20	1035	21	25.5	21		12	13.5	9.6	15.8

TABLE 14
(continued)
MEMBRANE FLUX BY STAGE DURING PILOT PLANT PROGRAM

FEED*	Date	Cumulative Hours of Operation	Permeate Flux by Stage, gfd							Average flux, gfd
			1a	1b	1c	2	3	4	5	
PCEF	2-21	1052	19.5	24	21		12	13.5	9.6	15.6
	2-22	1072	18.8	24.8	21		13.5	15	10	15.3
	2-23	1090	18	24	19.5		12	15	8.3	14.8
	2-26	1106	17.3	24	18.7		13.8	15	4.5	14.1
	2-27	1128	16.5	22.5	16.5		12	14.2	10	13.7
	2-28	1140	16.5	23.3	18		12	13.5	9	13.7
	3-1	1154	19.5	19.5	19.5		14.9	11.8		13
	3-2	1171	17.1	24.6	17.1		12.2	13.2		12.6

*FEED Abbreviations Key:

PCEF - Pine Caustic Extraction Filtrate
HDE - Hardwood Decker Effluent
PDE - Pine Decker Effluent
MDE - Mixed Decker Effluents

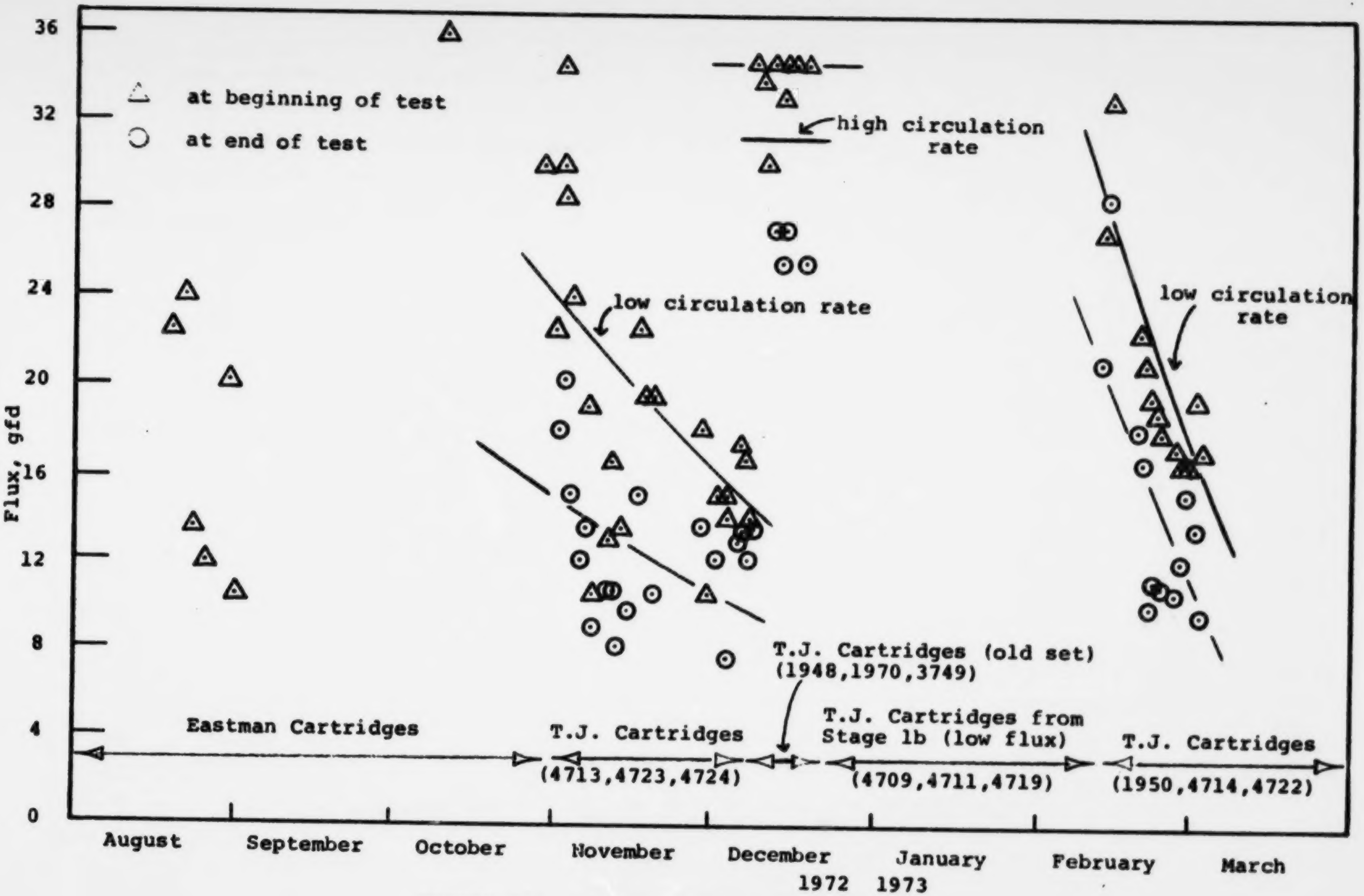


FIGURE 36: MEMBRANE FLUX FOR STAGE 1a

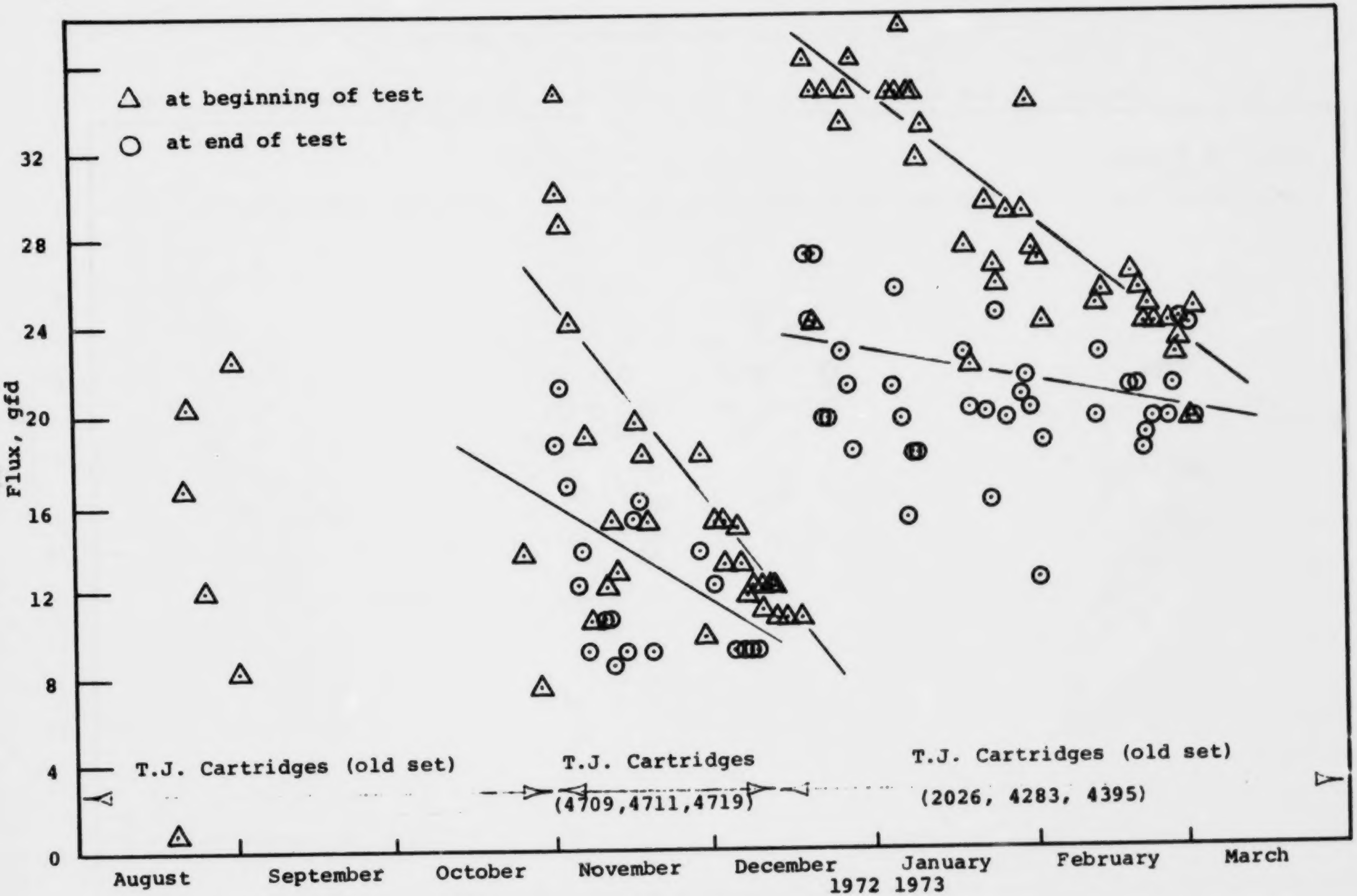


FIGURE 37: MEMBRANE FLUX FOR STAGE 1b

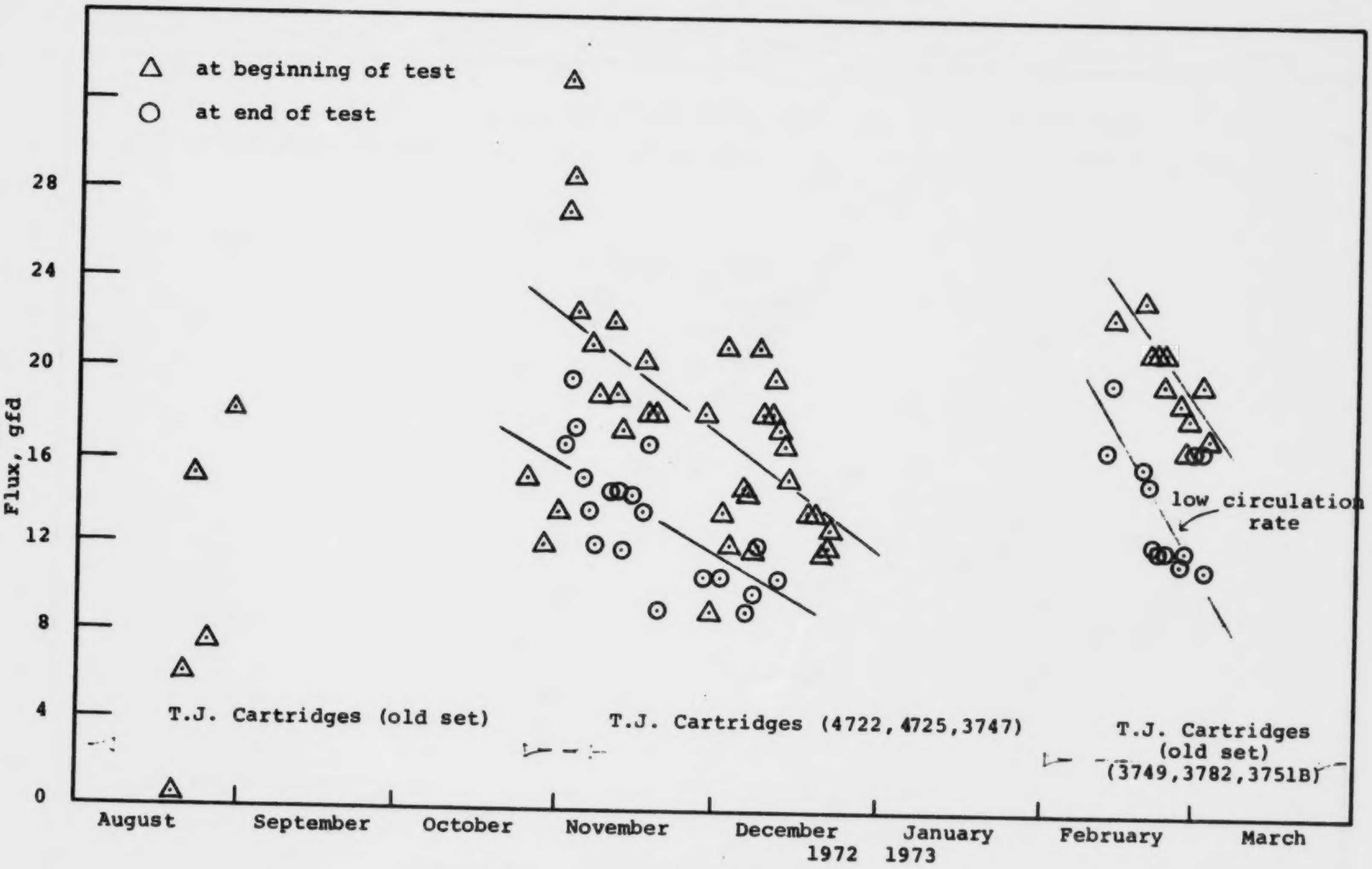


FIGURE 38: MEMBRANE FLUX FOR STAGE 1c

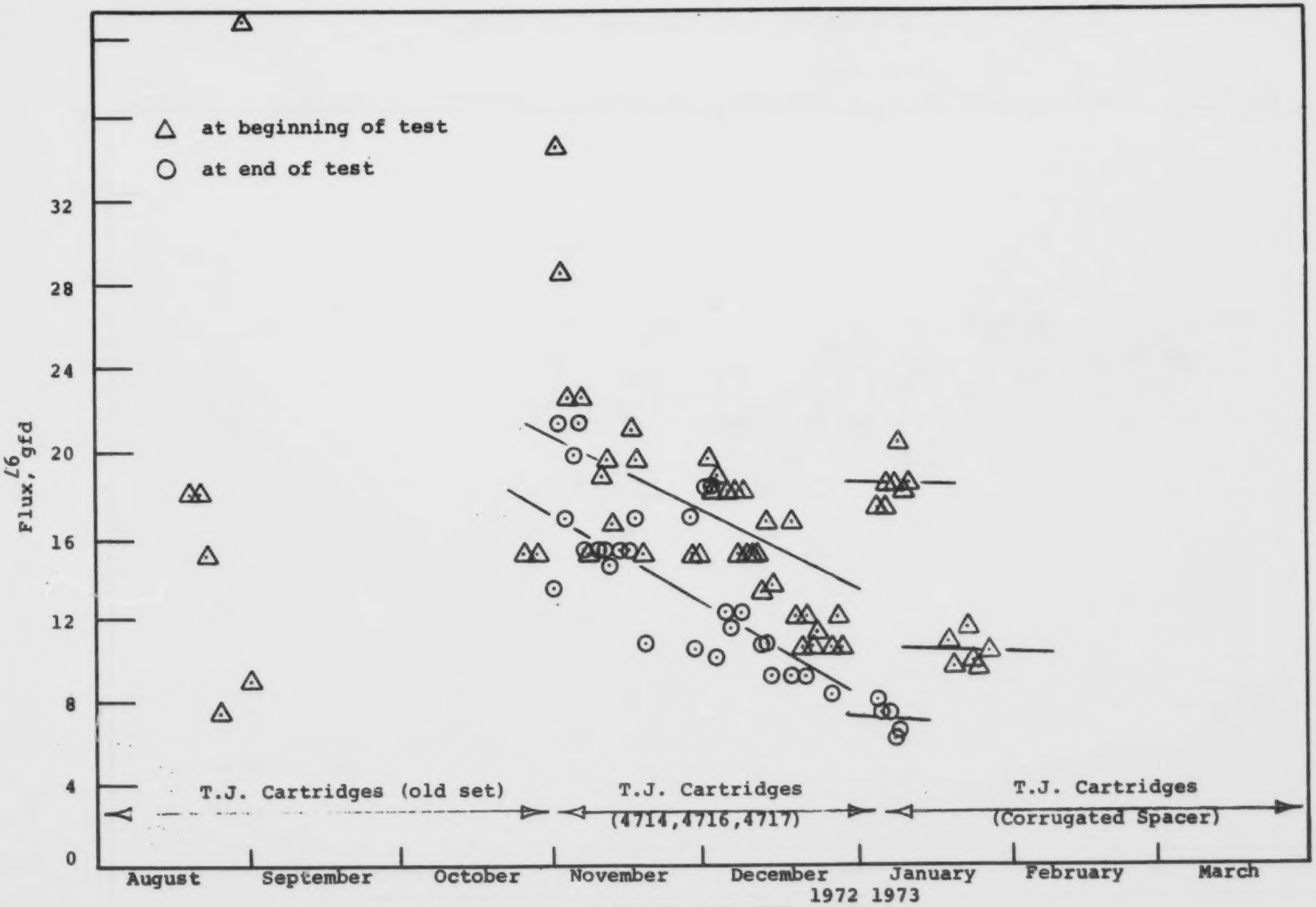


FIGURE 39: MEMBRANE FLUX FOR STAGE 2

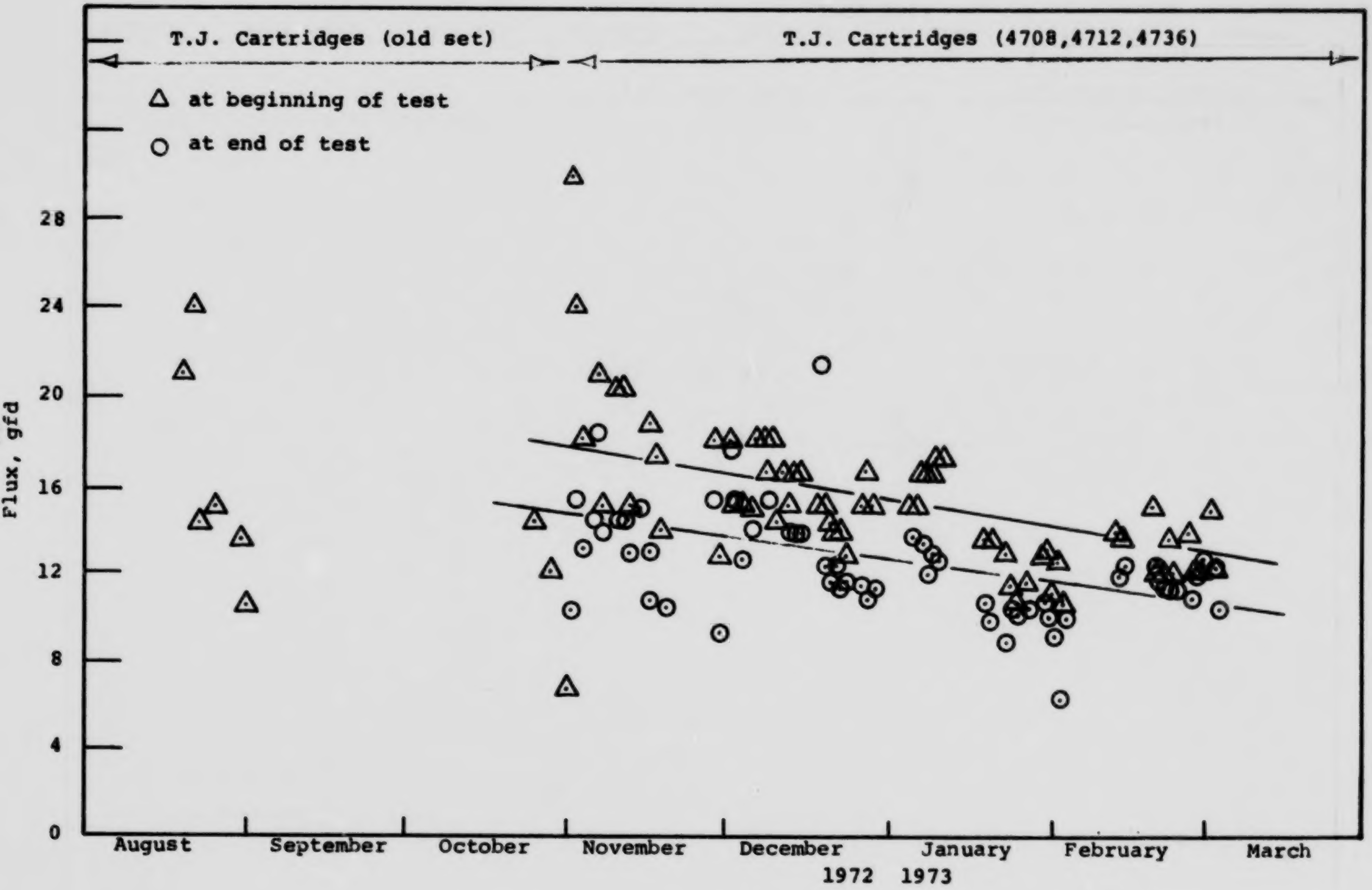


FIGURE 40: MEMBRANE FLUX FOR STAGE 3

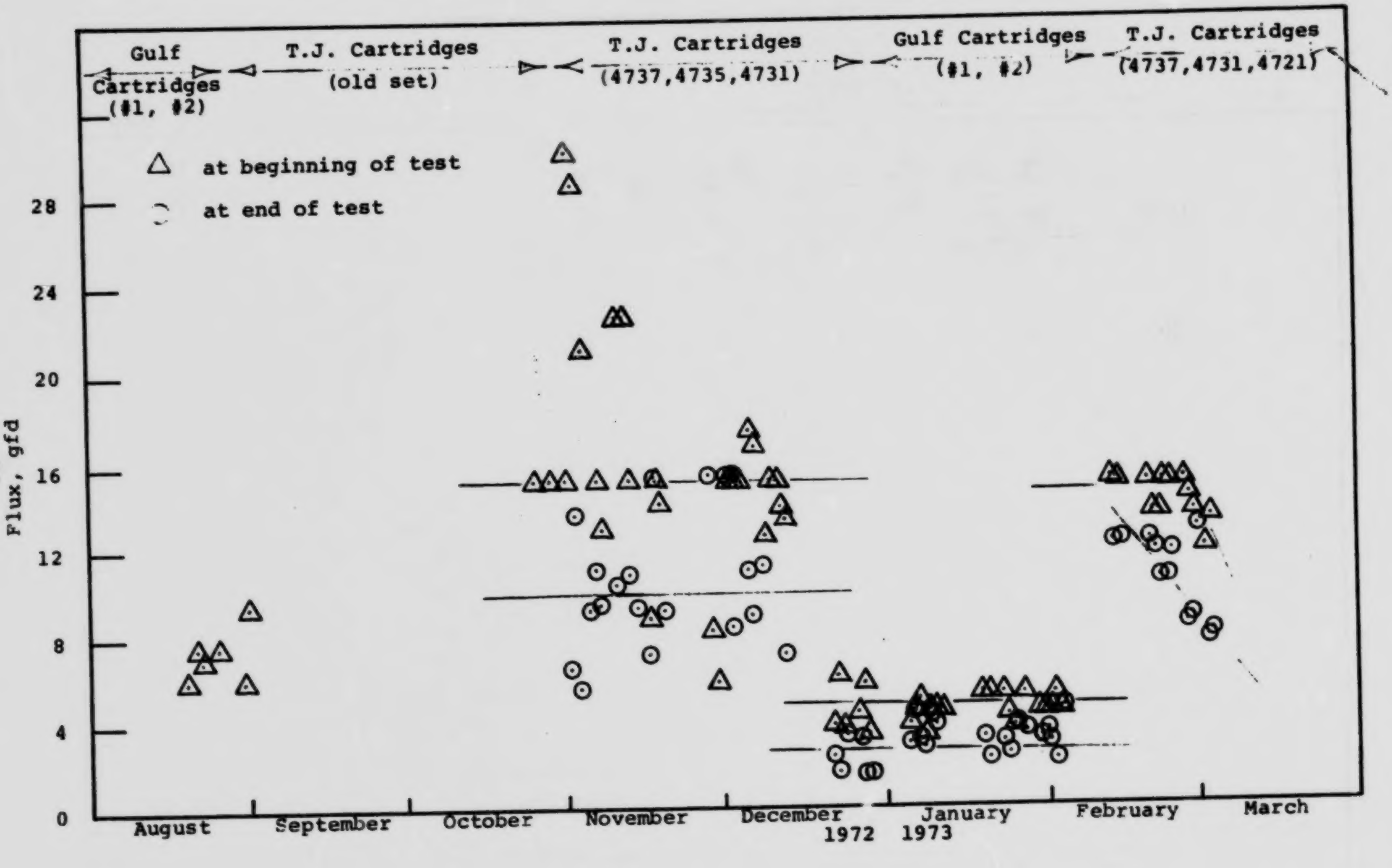


FIGURE 41: MEMBRANE FLUX FOR STAGE 4

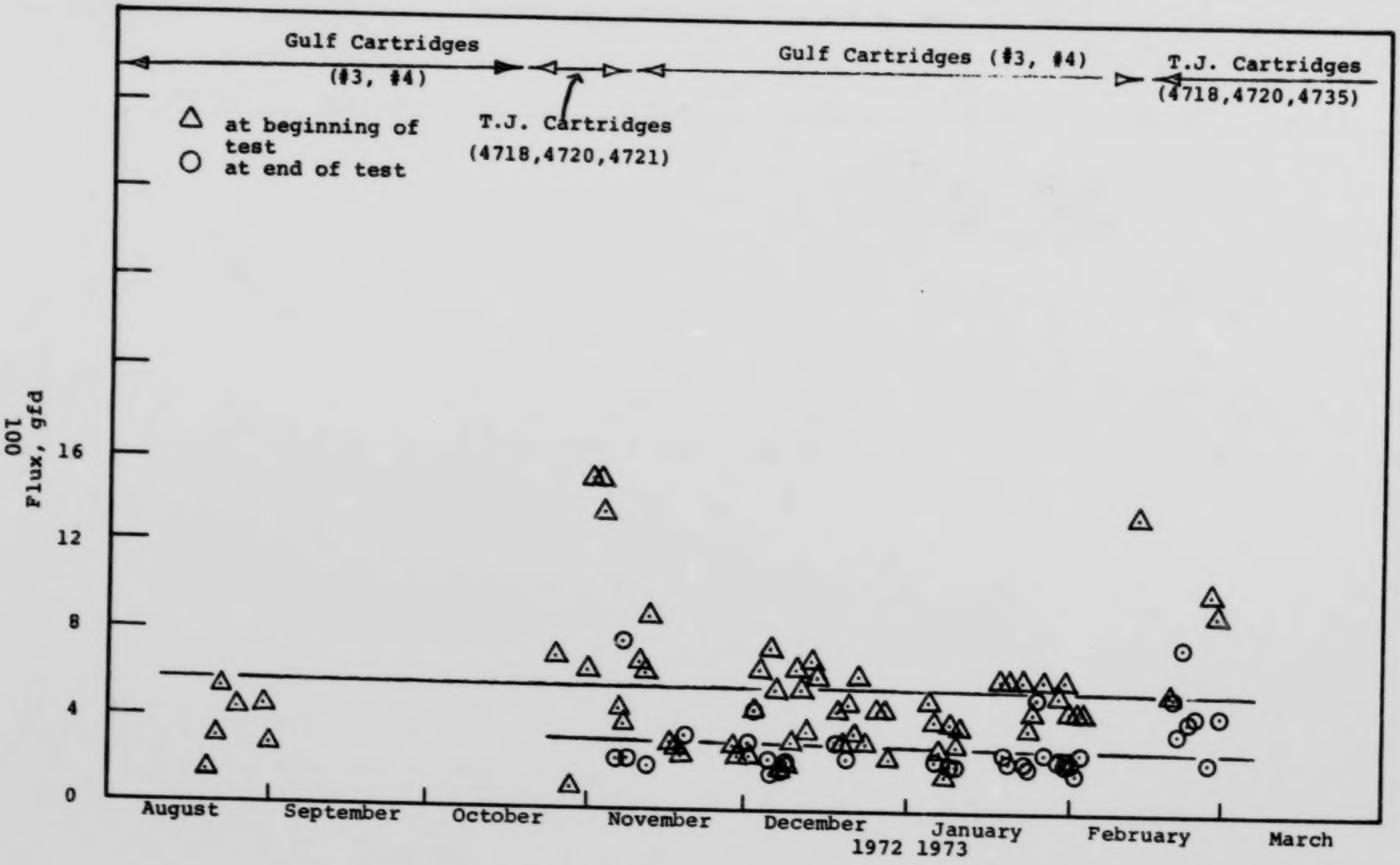


FIGURE 42: MEMBRANE FLUX FOR STAGE 5

1. Operating period: 8/19/72-8/31/72 (Start up). Pilot plant operation was initiated with pine caustic extraction filtrate on 8/19/72. At this time, the membrane system was equipped with Eastman cartridges in Stage 1a; T.J. Engineering cartridges in Stages 1b, 1c, 2 and 3; and Gulf cartridges in Stages 4 and 5.

It was immediately apparent that two related problems were present. First was that the ultrafiltration rate declined very rapidly during the course of an experiment due to membrane fouling. Second, restoring the flux by flushing or cleaning the membranes proved to be difficult. Subsequent runs showed the identical pattern of rapidly declining flux after start up, and great difficulty in membrane cleaning.

This behavior was evaluated, and the following conclusions were drawn:

- --the filtration system, consisting solely of the Broughton 10μ basket filters, was inadequate (as discussed in Section V.A.); and
- --the problem was possibly aggravated by the fact that under certain conditions it was possible for unfiltered feed to bypass the Broughton filters and enter the membrane system. This possibility was eliminated by installing a check valve in the appropriate place (Stage 1 recirculation loop) on 8/31/72.

On the basis of these results it was decided to evaluate other filtration alternatives to avoid or minimize the membrane flux decline. In order not to expose the entire membrane system to the possibility of inadequate filtration by the filtration sequences to be examined, it was decided to conduct these tests with a single membrane cartridge.

2. Operating period: 9/1/72-10/24/72 (single cartridge tests). A series of 50 to 100-hr tests were conducted with various filtration sequences and a single membrane cartridge. The first test was with the Broughton filters. Data for short term operation are contained in Figure 43. As is evident, there was a very rapid flux decline, which confirmed the conclusion that the 10μ Broughton filter was inadequate.

A more retentive filtration system was then installed. The new filtration sequence consisted of:

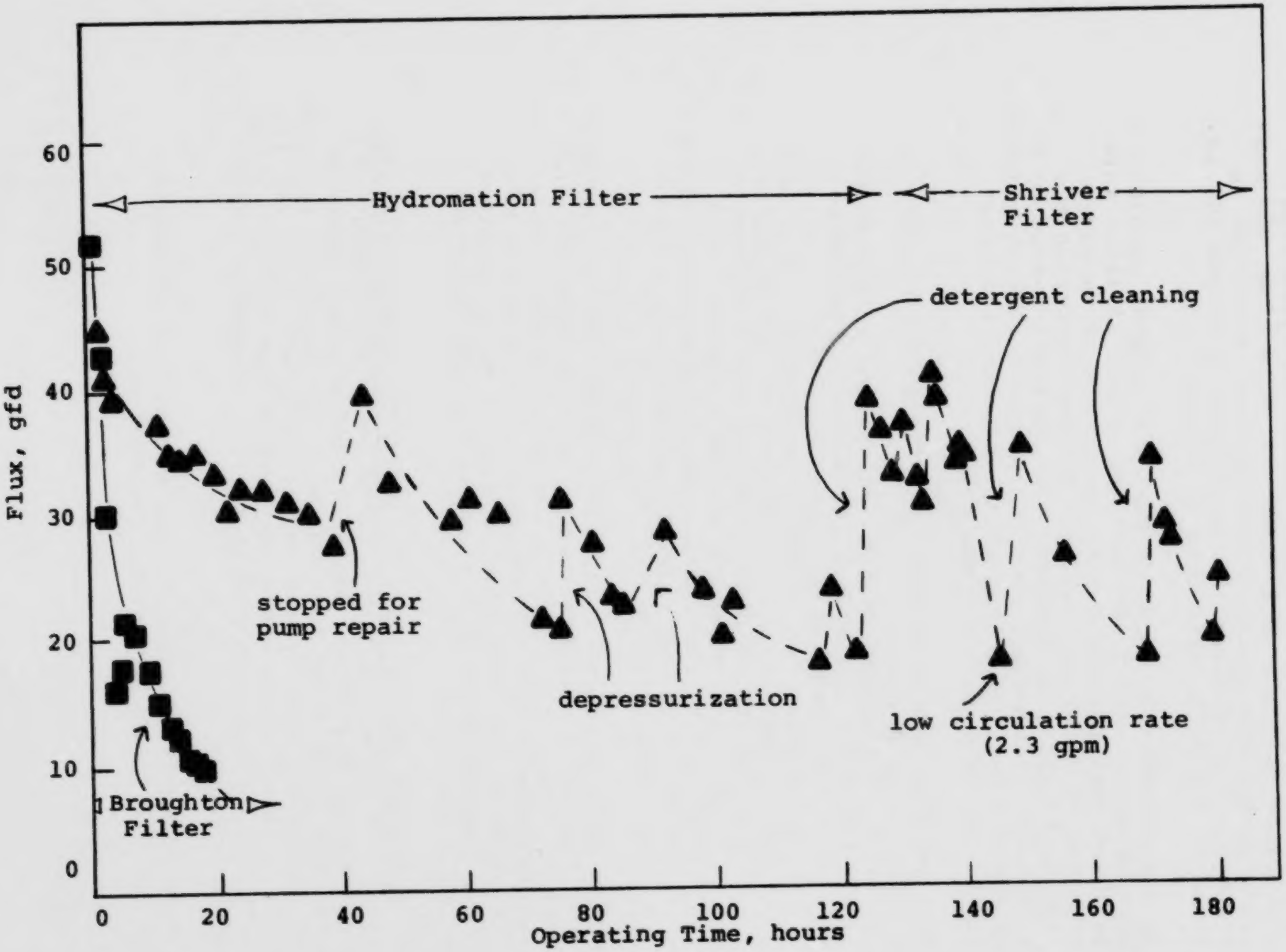


FIGURE 43: EFFECT OF PREFILTRATION EFFICIENCY ON ULTRAFILTRATION RATE
(T.J. Eng. Co. Cartridge, 110 psig; 103°F, 5 gpm circulation rate)

--a Bauer-hydrasieve filter (to remove fibers and gross solids);
--a Hydromation depth filter (as the main filter); and
--1μ Cuno cartridge filters (for final polishing).

The suspended solids removal efficiency of this filtration sequence was substantially better than that of the Broughton filters. This was reflected in a substantial improvement in membrane flux stability, as seen in Figure 43. It was possible to maintain a membrane flux exceeding 20 gfd with this filtration sequence during a 130-hr test. The short term flux decline (i.e. over 24 hrs) was about 25 to 35%, and it was possible to largely recovery flux by intermittent shut down (system depressurization). Complete flux recovery was achieved by detergent cleaning.

The cleaning procedure consisted of a low-pressure (20-40 psig) and high flow (5-6 gpm) once-through water flushing, and detergent cleaning (described in Section V.F.).

Unfortunately the filtration capacity of the Hydromation filter was less than that required to treat the feed needed for operation of the entire pilot plant. The filter vendor was unable to deliver a larger unit within the budget and schedule constraints of the program. Therefore it was decided to install a precoat filter, which was expected to give particulate removal equivalent to that of the Hydromation depth filter.

As described in the section on pretreatment, a Shriver filter press was available within the mill. This was installed in place of the Hydromation depth filter, keeping both the Bauer hydrasieve and the Cuno polishing filters in the system.

The single membrane cartridge was operated for about 50 hrs with the Shriver filter press, using wood flour as a precoat material and body feed. This filtration sequence was also found to effectively remove suspended solids as shown by the data in Figure 43. Its filtration efficiency was somewhat less than that for the Hydromation filter, since the rate of membrane flux decline within relatively short periods, e.g. 24 hrs, was somewhat more rapid. However, it appeared that acceptable ultrafiltration rates could be maintained, and membrane cleaning efficiency was greatly improved. Moreover, the Shriver filter press had sufficient capacity for operation of the entire pilot plant. Thus at the end of these tests with the single cartridge it was decided to resume operation of the entire pilot plant.

3. Operating period: 10/25/72-10/31/72 (renewed pilot plant tests). The pilot plant operation was renewed on 10/25/72 using the Shriver filter press. The first step was to clean the pilot plant according to the detergent cleaning method developed for the single cartridge. The result was a substantial improvement in flux for all stages (see Table 14). However, during operation on subsequent days, the flux decline was unsatisfactory. This was particularly true for Stages 1b, 2 and 3. At the time it was concluded that the initial set of membrane cartridges was irreversibly fouled or plugged by inadequately filtered feed during August, and was not suitable for obtaining meaningful results. A new set of membrane cartridges was ordered, in order to conduct the remainder of the experimental program under representative conditions, that is, with membrane cartridges which had not been subjected to extreme upset conditions. It now appears likely that the unsatisfactory flux decline observed at the end of October was due to a combination of this hypothesis and inadequate filtration with the Shriver filter press.

4. Operating period: 11/1/72-11/27/72. The new set of T.J. Engineering cartridges was received and installed in the pilot plant on 11/1/72. Operation was resumed with pine caustic extraction filtrate. Initial fluxes were high, in the range of 25-35 gfd. By reference to Figures 36 to 42 and Table 14, it can be seen that performance was substantially improved over that of any earlier period. Nevertheless, a continuous decline in flux and increase in pressure drop across the membrane stages was observed. Complete flux recovery by the cleaning procedure developed for the single spiral was not obtained. However, the system appeared to be stabilizing at a flux rate level of approximately 15 gfd, but pressure drops in Stages 1, 2 and 3 were high.

One membrane change was made during this period. When a leak occurred in Stage 5 on 11/6/72, the cartridges were replaced with two of the original Gulf membrane cartridges. These cartridges remained in the pilot plant until 2/12/73.

During this start up period with the new membranes several observations were made on the condition of the cartridges. These were:

Date	Observation
11/14/72 to 11/17/72	Brine seals of almost all cartridges, except the two Gulf cartridges in Stage 5, were found to have "flipped", potentially allowing feed to short-circuit the membranes. Membrane cartridge compression effects were also noticed (see Section V.G.: Module Mechanical Problems). The brine seals were reinforced and the cartridges returned to their respective shells.
12/7/72	The brine seals of the cartridges in shells la, lb, and lc had flipped. The cartridges were reinstalled with the brine seals properly positioned.

In addition, other limitations of the system became apparent during this period. Specifically, the pilot plant piping did not permit once-through flushing of the membrane cartridges at low pressure and high flow. Also, it was suspected that the Shriver filter press was not providing a high efficiency for suspended solids removal.

Thus, the following system modifications were undertaken and completed by 12/7/72.

- --A continuous wood flour body feed addition system was installed.
- --A regular procedure was instituted to apply a layer of fine paper on top of the filter cloth of the Shriver filter press. This aided in the formation of the precoat layer, as well as facilitating filter cleanup.

--Piping modifications were made such that each membrane shell could be flushed at high flow and low pressure on a once-through basis.

5. Operating period: 11/28/72-1/10/73. After the changes to the filtration system and piping had been made, operation continued on a regular basis. Additional membrane changes were made, as summarized below.

Date	Change
12/8/72	Stage 1a cartridges leaked; replaced with three TJ Engineering cartridges from the initial set.
12/19/72	Switched position of Stage 1a and 1b cartridges.
12/21/72	Stage 1b(formerly 1a) cartridges were replaced by three TJ cartridges from the initial set.
12/21/72	Installed two Gulf cartridges from the initial set in Stage 4.
1/4/73	Installed three TJ Engineering wide-channel corrugated-spacer cartridges in Stage 2. Observed that two of the three cartridges removed had torn brine seals.

Stage 1

On 12/8/72, Shell 1a was observed to have a leak. The membrane cartridges were replaced with three of the original set of TJ Engineering cartridges. Surprisingly, these three older cartridges showed a very high flux, about 30 gfd.

To determine if the position of the 1a Shell influenced performance, the three cartridges in Stage 1a were shifted to the 1b position on 12/19/72. For the two days during which these cartridges were tested in this position, membrane flux continued to be high.

On 12/21/72, the three cartridges in the 1b position were replaced by another three TJ Engineering cartridges from the initial set, in order to see if they also had high flux. As seen from the data in the Table 14, these three cartridges did indeed exhibit high, stable flux, and continued to do so for the rest of the test program. Specifically, flux remained more or less constant until 1/10/73, when operation with decker effluents was initiated.

It was immediately suspected that unequal flow distribution between the parallel shells in Stage 1 caused this behavior. Just before beginning the tests with the decker effluent, flows through the shells in Stage 1 were measured. It was observed that approximately 65% of the total flow was passing through Stage 1b, 30% through Stage 1a, and only 5% through Stage 1c.

It was concluded from these results that it is necessary to maintain a high flow through spiral-wound cartridges with standard mesh spacers to maintain a satisfactory flux. High flow also results in facilitated cleaning of the membrane cartridges and flux recovery. Furthermore, in an actual plant installation, flow distributors between parallel shells will be required. Finally, it appears that a minimum circulation rate of about 8 gpm will be required to maintain high flux.

Data showing performance of these membrane cartridges operating under acceptable conditions are contained in Figure 44. Shown is flux as a function of operating time. During any period between cleaning, flux decreased due to reversible membrane fouling. However, membrane flux was completely recovered during cleaning.

Stage 2

Stage 2 fluxes ranged between 15 and 22 gfd until mid-December. After this time, flux began to decline slowly. Pressure drop for Stage 2 increased to a level of about 70 psi (at 4.5 gpm circulation rate) on 11/29/72. After-

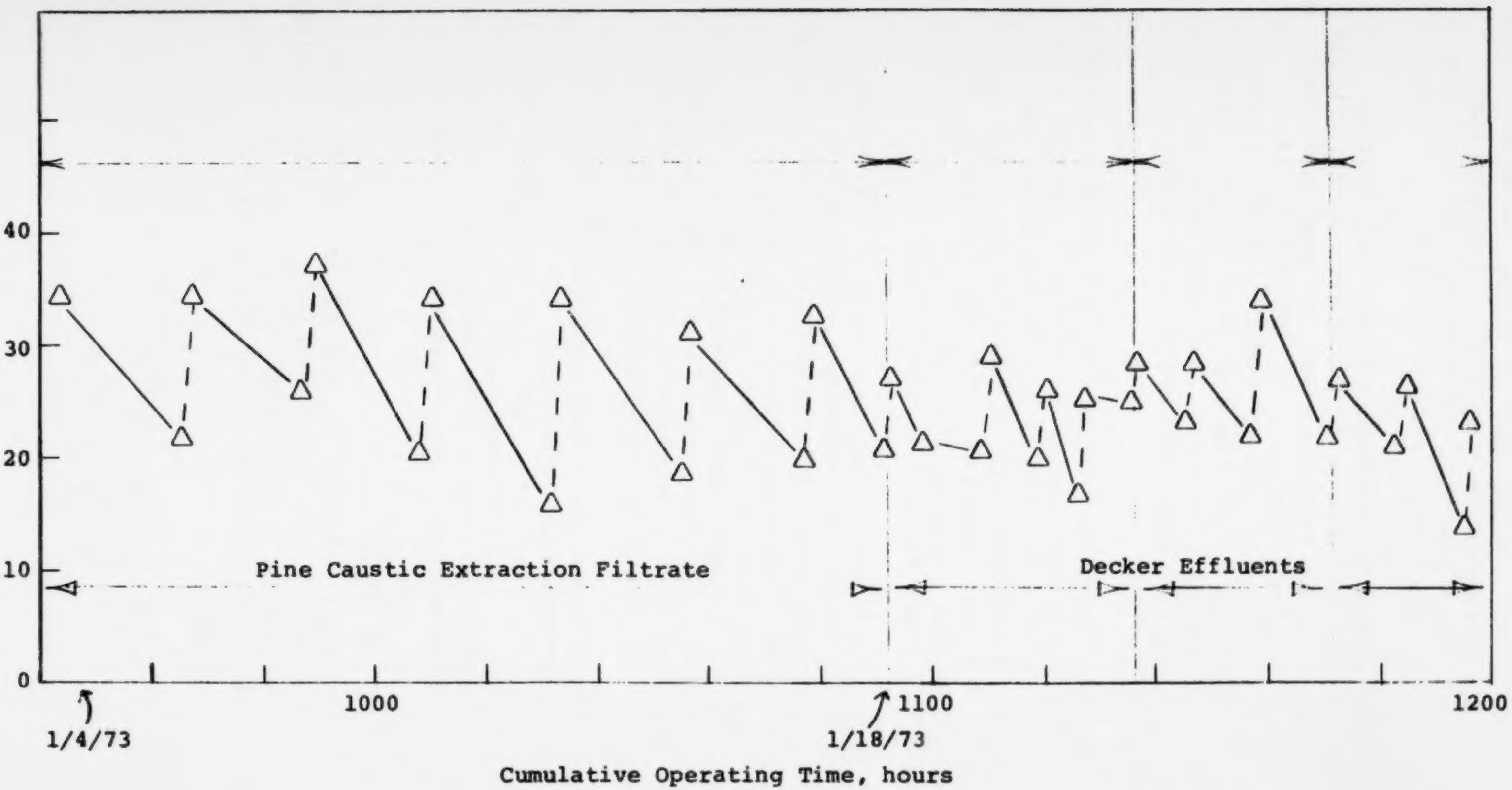


FIGURE 44

ULTRAFILTRATION RATE DATA: STAGE 1b

wards, pressure drop decreased and this was due to brine seal reversal. On 1/4/73, when the Stage 2 cartridges were removed to replace them with three new TJ Engineering cartridges (wide-channel, corrugated spacers), it was observed that two out of the three Stage 2 cartridges had brine seal failures. It is likely that after brine seals failed, feed partially bypassed the cartridges which then slowly fouled due to low flow (see Table 14).

Three corrugated spacer cartridges were installed in Stage 2 on 1/4/73 to determine if this "hydrodynamically clean" flow channel spacer would permit operation without cartridge plugging. It is generally necessary to operate this type of cartridge at a higher feed flow rate than in mesh spacer cartridges. The open cross-sectional area in corrugated space cartridges is greater, and unless one operates at the same or higher linear velocity than in a mesh-spacer cartridge, concentration polarization can reduce flux below the water flux of the cartridges. It was anticipated that the flow rate required to overcome this effect of concentration polarization would exceed the 5-6 gpm which the Stage 2 circulation pump could deliver. Thus, the purpose of the test was not to collect meaningful ultrafiltration rate data but to demonstrate that the cartridges could operate without flow channel blockage and the corresponding irreversible buildup in pressure drop and flux decline.

An examination of the data in Figure 45 shows that no long-term degradation in flux was obtained in the tests with the pine caustic extraction filtrate. (The data for the decker effluents are explained below). The clean module rates, were observed to be about 50% of the water flux of the cartridges, and this demonstrates that concentration polarization was relatively severe. By contrast, flux of pine caustic extraction filtrate in all other stages was generally observed to be identical to the water flux.

Since the initial flux of the corrugated spacer cartridges could be recovered repeatedly, it is concluded that the use of this flow channel spacer can result in complete membrane cartridge cleanup.

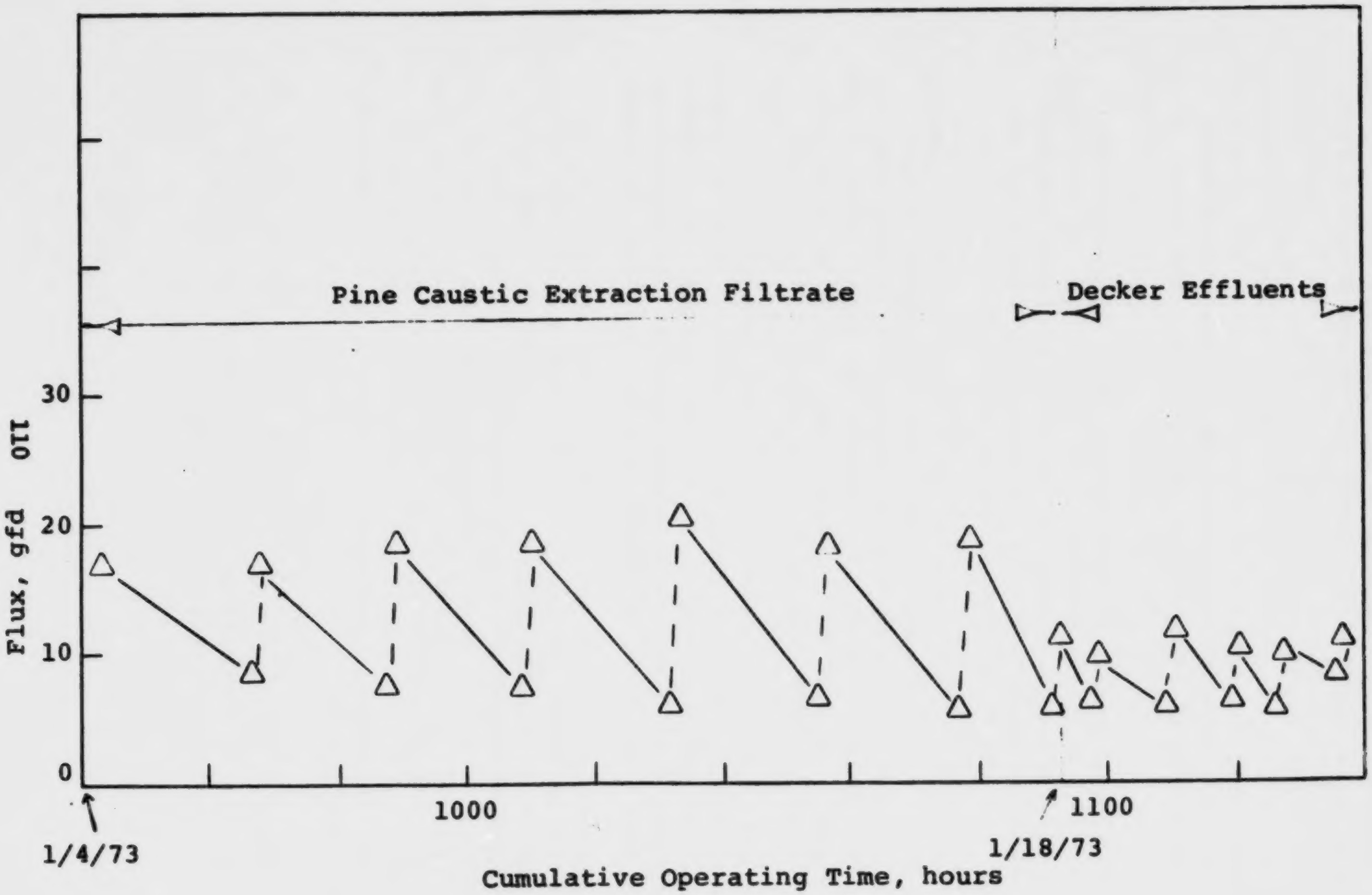


FIGURE 45. ULTRAFILTRATION RATE DATA: STAGE 2

Stage 3

The membrane flux of the Stage 3 membrane cartridges remained between 12 and 18 gfd througout this test period. It is concluded that neither brine seal failure nor substantial irreversible flow channel plugging occurred. The gradual decline in flux is attributed to irreversible membrane compaction (see Section V.F. for the effect of time on water flux of these membranes).

Stages 4 and 5

The flux of Stage 4 also remained reasonably high, until 12/12/72, at which point a leak occurred and flux was no longer measured.

Membrane flux for the Gulf cartridges in Stages 4 and 5 generally ranged between 2 and 6 gfd, and this is attributed to the lower flux characteristics of the Gulf membranes. It is also possible that these membrane cartridges may have partially dried out during the period that they were not used.

6. Operating period: 1/18/73-2/2/73 (Operation with Decker Effluents). During the end of January, 1973, the pilot plant was operated for periods of approximately 40 hrs with hardwood and pine decker effluents. Unfortunately, these tests were conducted when the pilot plant system was not operating with acceptable flux in most of the stages. Thus, the tests with the decker effluents provide preliminary information on the separation efficiency of the membranes and, to a limited extent, values of membrane flux.

Referring to Table 14, meaningful flux can be obtained from the data for Stages 1b, 2 (corrugated spacer cartridges), and 3. The data for Stage 1b and 3 indicate that the flux for the two decker effluents was somewhat less than flux for the pine caustic extraction filtrate. This was more pronounced for Stage 2. This behavior is explained by the following hypothesis. The organics contained in the decker effluents have higher molecular weights than those in the pine caustic extraction filtrate, since fragmentation of the organics in the latter waste occurs in the bleach plant chlorination stage. Thus, at a given retained organics loading, concentration polarization should be more severe for the decker effluents since the diffusivity of the dissolved solutes is lower. For this reason, flux in Stage 2 was reduced by almost a factor of two below that for pine caustic extraction filtrate.

The lower flux in Stage 1 may also be due to a gradual failure of the Stage 1 pump. It was discovered that the capacity of the Stage 1 pump was low and this could have resulted in a flux reduction due to concentration polarization. Also, near the end of the tests with the decker effluents, the Stage 2 pump began to fail, and this may account in part for the reduced Stage 2 flux.

7. Operating period: 2/3/73-3/1/73. At the end of the tests with the decker effluents it was apparent that additional modifications to the pilot plant were desirable. The following changes were made:

--a Sparkler pressure leaf filter replaced the Shriver filter press;
--a Sparkler-Velmac 5μ disc filter (backwashable) replaced the Cuno cartridge filters;
--the Stage 1 and Stage 2 pumps were repaired, and their flow capacity was restored.

During the first week in February, 1973, all the TJ Engineering cartridges were tested in the single cartridge test shell. On the basis of the results of these tests, the best membranes were selected and installed in the pilot plant. Details are given in Appendix B. Briefly, the cartridges in Stages 1b, 2, and 3 were not changed; the four Gulf cartridges in Stages 4 and 5 were replaced by six TJ Engineering cartridges; two of the three cartridges in each stage had been previously used in the same stage in November, 1972. Stages 1a and 1c were filled with other good remaining TJ Engineering cartridges.

Initially some persistent problems with O-ring leaks were encountered, but eventually these were solved. Operation began on 2/12/73 with pine caustic extraction filtrate. In general, membrane fluxes were similar to those observed in November and December.

The membranes in Stages 1b and 3 had not been changed. In treating pine caustic extraction filtrate, the flux for Stage 3 returned almost to the original level. But in the case of 1b there was a certain amount of irreversible flux loss. The reason for this is not clear.

In Table 14 flux levels are shown for both the beginning of an experiment (approx. 1 hr) and the end (usually 8 to 17 hrs). The flux decline for Shell 1b usually did not exceed 20%, compared to declines of 40 to 50% for Shells 1a and 1c. This is presented graphically in Figure 46, which contains data for Shells 1a and 1b. It is seen that the initial water flux for the two shells were identical. However, the initial flux with pine caustic extraction filtrate was higher for Shell 1b and did not fall off as rapidly as that of Shell 1a. Furthermore, membrane cleaning recovered the initial flux for Shell 1b, while for Shell 1a the initial flux decreased with time. In addition, the water fluxes of the two stages diverged, with water flux for Shell 1a declining relative to that for Shell 1b.

At this point, circulation flows in the different shells were measured and it was found that the flow through Shell 1a was approximately 1.3 gpm; Shell b, 9.5 gpm; and Shell 1c, 4 gpm. So again it was apparent that the shell with the highest circulation flow exhibited superior performance.

In general, flux for the pine caustic extraction filtrate was equal to the water flux of the membranes. Notable exceptions were the behavior in Stage 2, with corrugated spacer spirals, and in Stage 5. As can be seen in Figure 42, the Stage 5 flux for the TJ Engineering cartridges was more or less equal to the Stage 5 flux for the Gulf cartridges, even though the TJ Engineering cartridges had a substantially higher water flux. This demonstrates that at least in Stage 5 the feed concentration attained a level sufficiently high to reduce flux, that is, flux in this stage was limited by concentration polarization, and not by the membrane water flux.

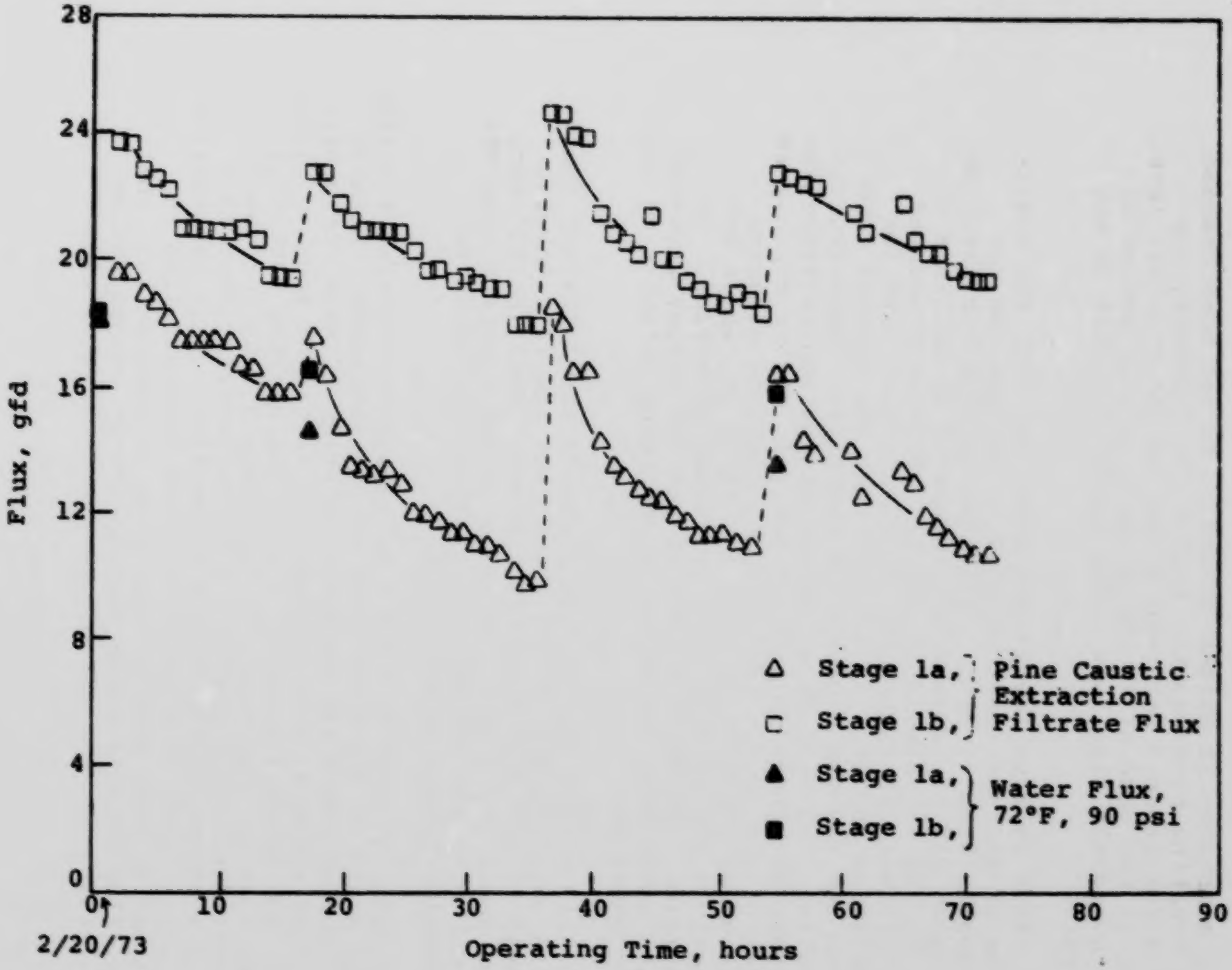


FIGURE 46: FLUX VS TIME FOR STAGES 1A and 1B

Since the color rejection of the TJ Engineering cartridges was lower than that of the Gulf cartridges (more color was observed in the Stage 5 permeate) it would be desirable in a full-scale installation to use lower flux, more selective membranes in the final stages).

D. PRESSURE DROP DATA

Pressure drop (and circulation rate) data for the five stages are shown in Figure 47 to 51. Pressure drop was strongly dependent on circulation rate, as can be seen from inspection of the data. For example, when the Stage 1 pump lost capacity (flow), pressure drop across this stage decreased sharply. (Note: one would expect turbulent-flow pressure drop to increase with the 1.8 power of circulation rate.)

During November, pressure drops for all stages became quite high, exceeding 50 psig in the early stages. In some stages (e.g. Stage 2), brine seal failure led to a reduction in pressure drop since feed flow bypassed the membrane cartridges.

The high pressure drop was due to inadequate prefiltration and an inability to flush and clean the cartridges thoroughly.

The changes made in the pilot plant in early December, piping modifications to permit once-through flushing and the use of paper on the plates of the Shriver filter press, resulted in improved membrane cleaning. This was generally reflected in lower pressure drops in the pilot plant after mid-December. The high pressure drop in Stage 1 in February, 1973 was due to operation at increased flow.

Pressure drop across Stage 2 when corrugated spacer cartridges were used was negligible, and no increase with time was observed.

Thus, it is concluded that negligible flow-channel plugging occurred with the corrugated-space cartridges.

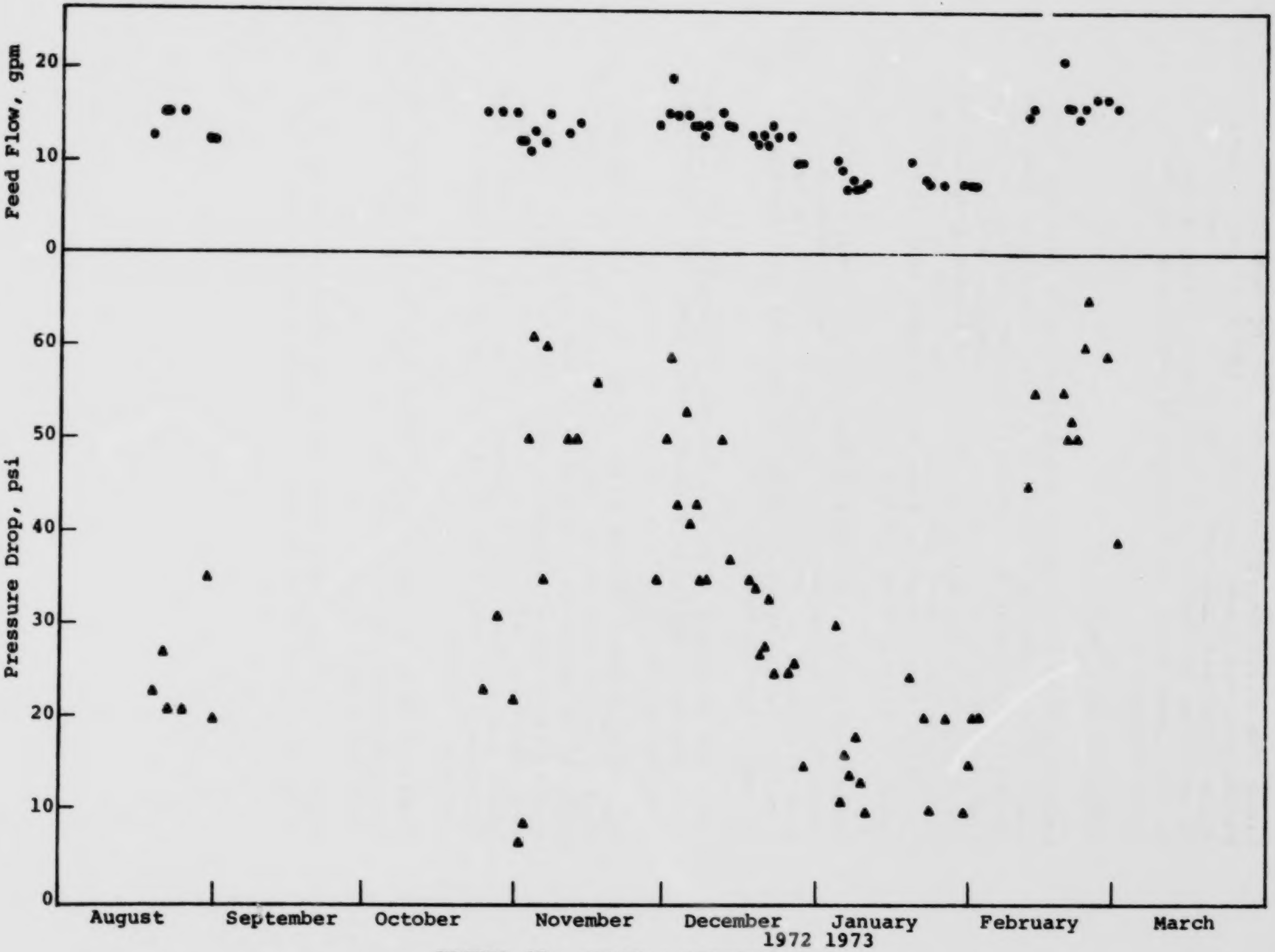


FIGURE 47: STAGE 1 PRESSURE DROP

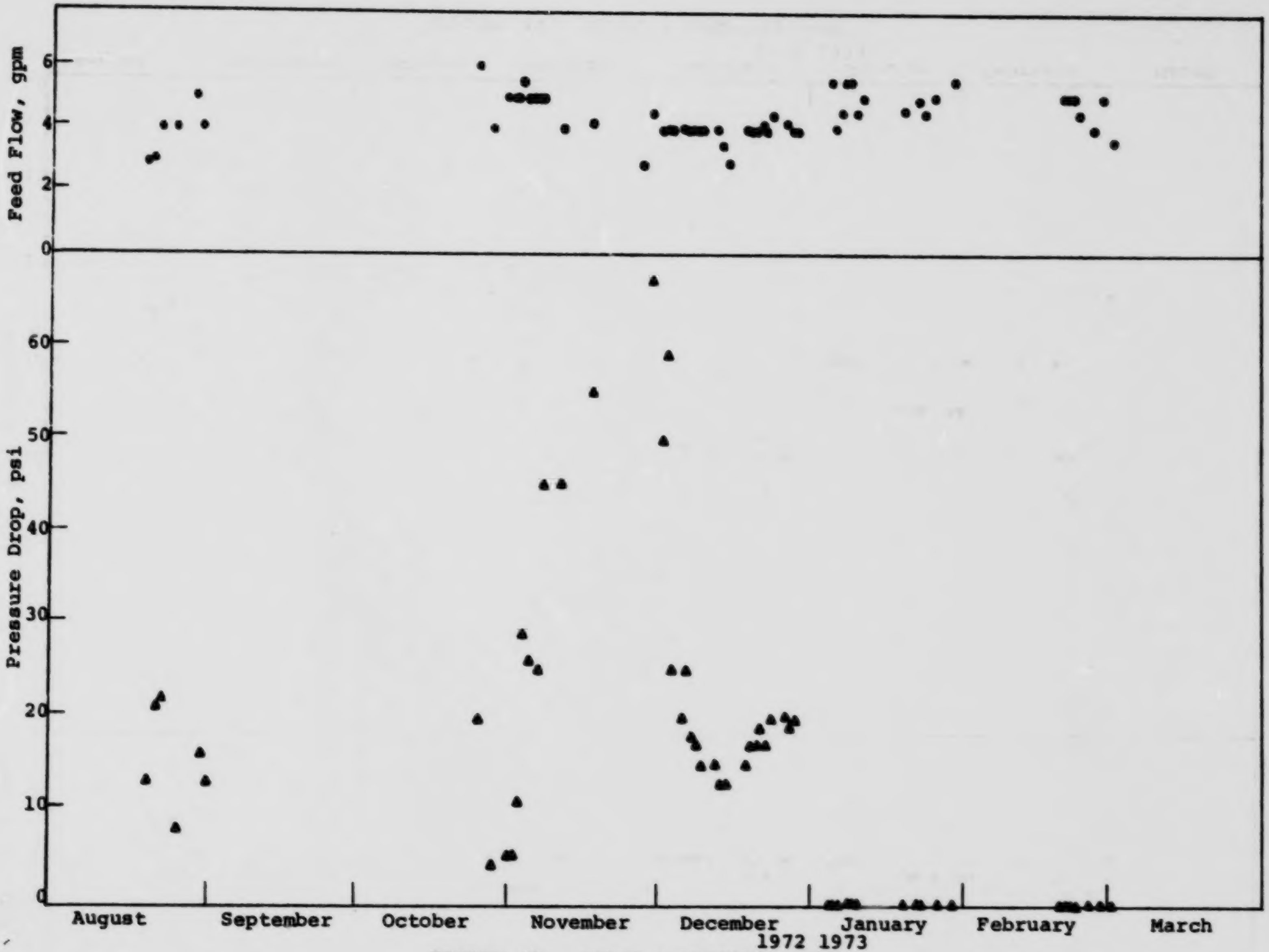


FIGURE 48: STAGE 2 PRESSURE DROP

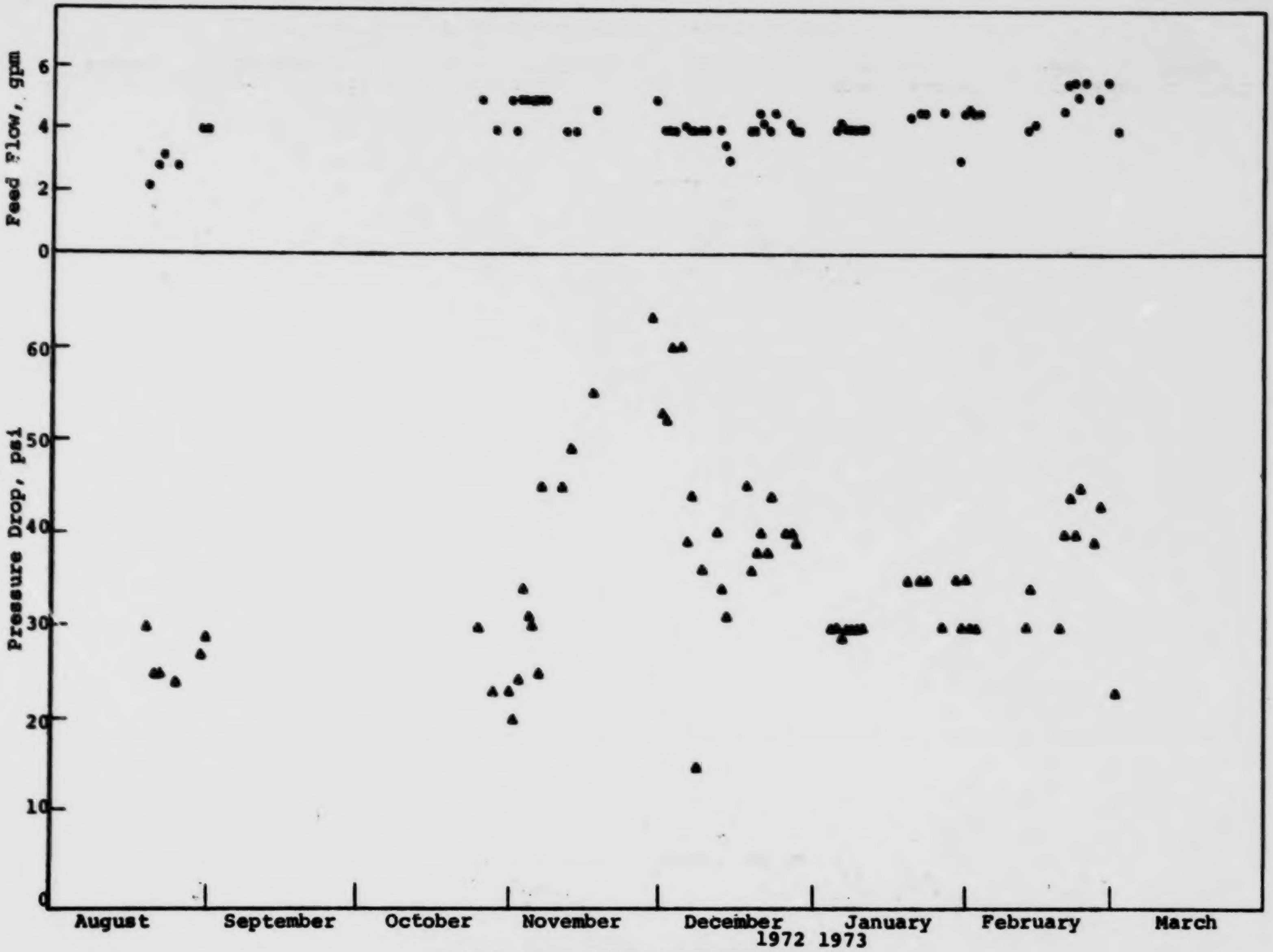


FIGURE 49: STAGE 3 PRESSURE DROP

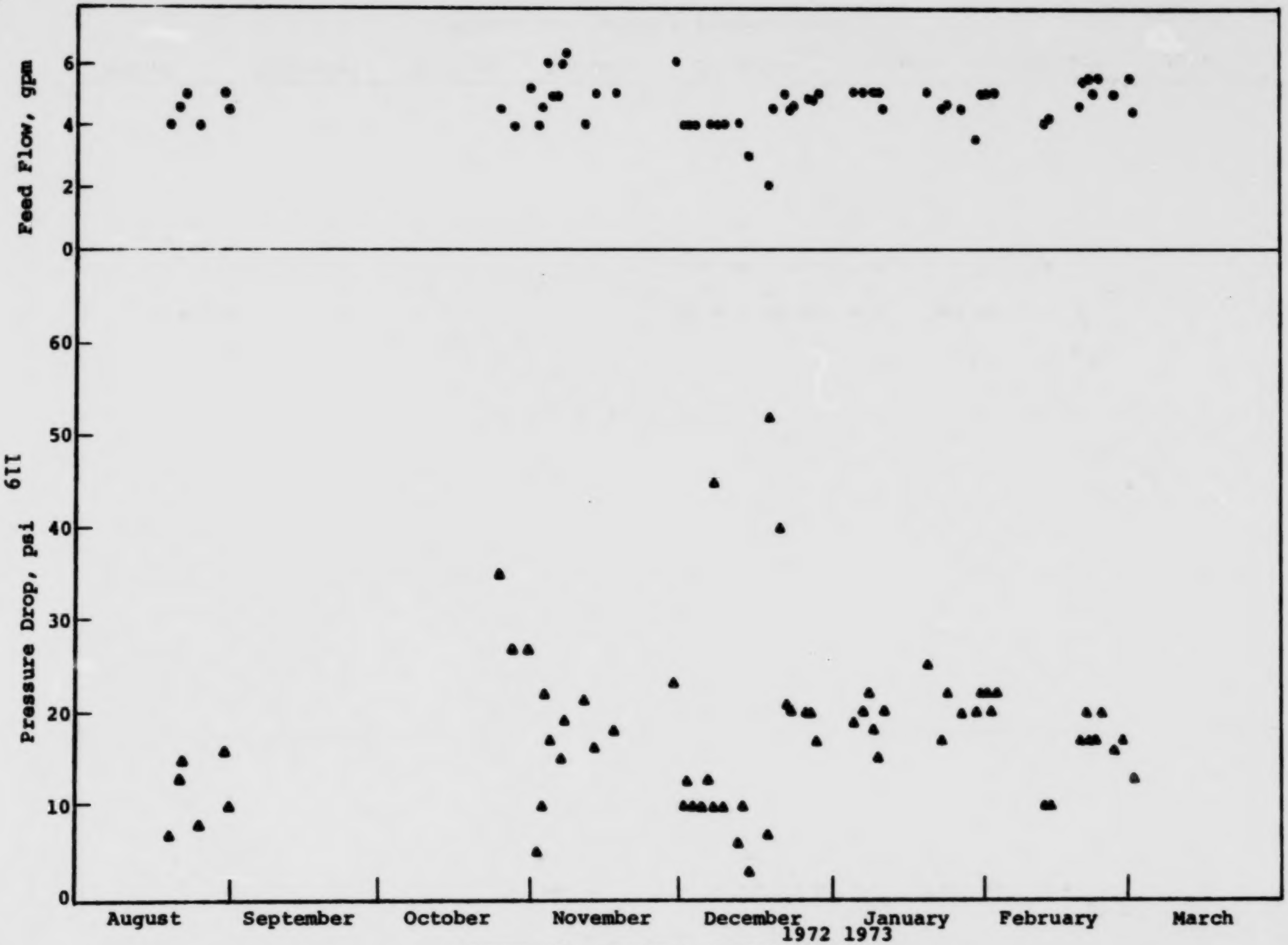


FIGURE 50: STAGE 4 PRESSURE DROP

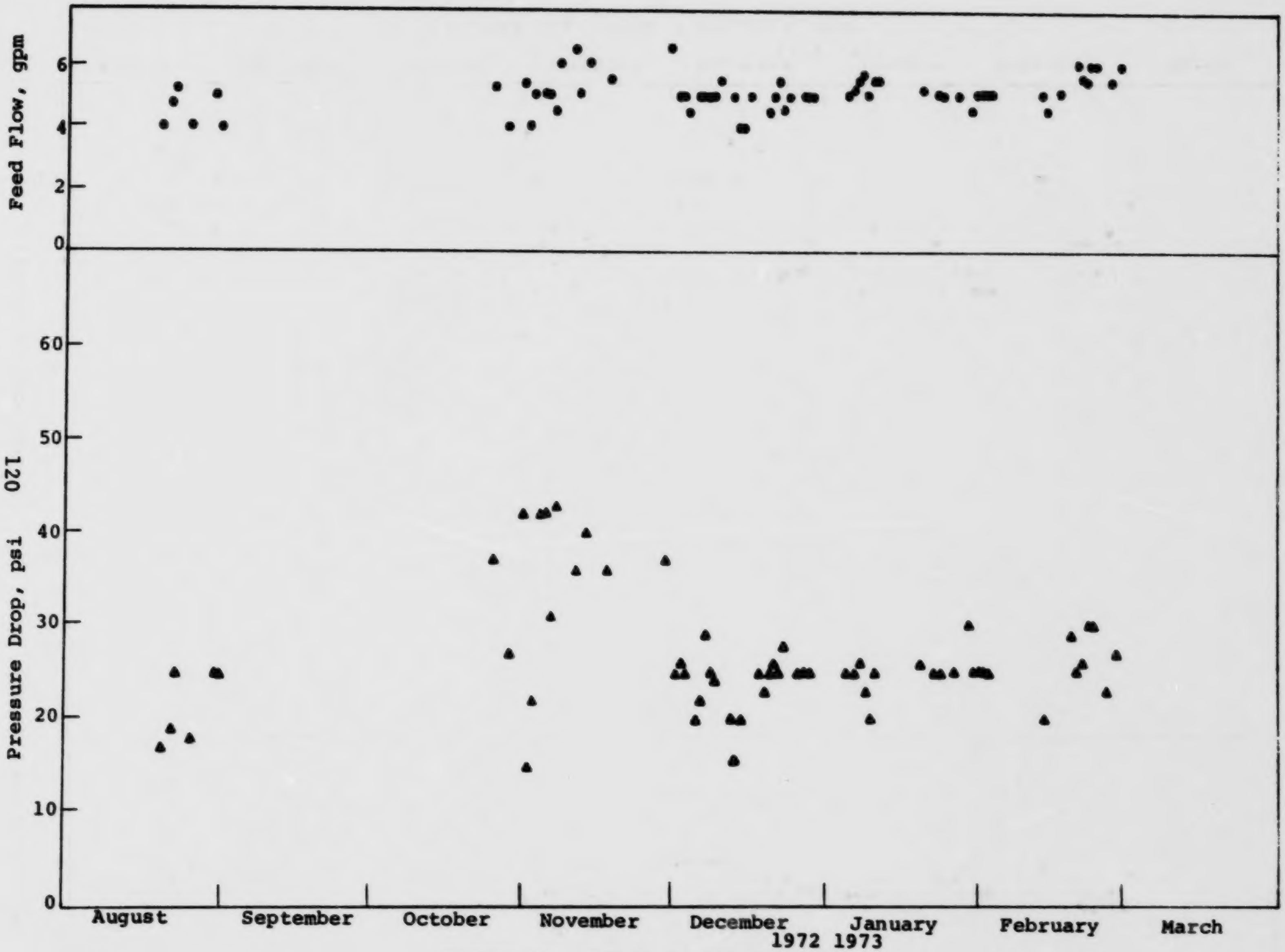


FIGURE 51: STAGE 5 PRESSURE DROP

E. IMPORTANT FACTORS CONTROLLING MEMBRANE FLUX

1. Slime Formation on the Membrane Surfaces

The membrane flux decline observed was controlled by several interrelated phenomena. It is instructive first to consider a "normal" case, in which a continuous and uniform feed flow is maintained across the membrane surface. In this case, a "gel" layer of slightly soluble colloidal and macromolecular solutes can build up on the membrane surface with time, reducing flux. This behavior has been reported previously for a variety of feed solutions (27), and undoubtedly occurred in the ultrafiltration of the three effluents processed in this program.

During the program "slimes" removed from membrane spirals were analyzed, and other tests were performed in an attempt to characterize the troublesome "foulants".

Analyses of the slimes indicated the presence of large amounts of ash in the slime (60-70%). The ash consisted primarily of finely divided clay, calcium compounds and anti-foam agents such as silicones. These ash materials were traced to paper machine white water which was used for a time as make-up for the caustic extraction stage. Beginning in January white water was not used for this function.

Materials similar in composition to the slime are found in the effluents of every pulp and paper mill process using water which has been in contact with cellulose pulp. In all cases, these effluents can be filtered (or even ultrafiltered), and the filtrate when refiltered 24-72 hours later will have $10\text{-}100^{+}$ppm of a grayish floc which appears similar to the material removed from the membrane surfaces. This suggests that the minimal membrane fouling and slime formation observed in earlier laboratory experiments may have been a result of the age of the material studied. The slime-forming material may have flocculated during the time of sample shipping and most of it may have been removed in the laboratory prefiltration.

In order to develop a better understanding of the mechanism of slime formation, a series of tests and analyses were conducted. The slimy solids were examined bacteriologically in several laboratories. No evidence was developed to indicate the presence of any microorganism.

An extensive experiment was conducted on fresh pine caustic extraction filtrate. The fresh sample was adjusted to a given pH. Suspended solids were determined by filtration of 500 cc samples through Whatman No. 40 filter paper. The filtrates were allowed to stand for 24 hrs at room temperature and at 120°F. The suspended solids in these filtrates were then determined as before.

A typical set of data from these experiments is given below:

Pine Caustic Extract Sample 12/7/72

Adjusted pH	Suspended Solids, ppm	Suspended Solids in Filtrate after 24 hrs, ppm at Room Temperature	
			at 120°F
(original) 12.5	53	4	2
8	43	2	2
7	39	4	1
6	58	29	28
5	113	41	43

From experiments of this type it appears that part of the problem of prefiltration and membrane fouling may be due to pH adjustment to pH 7 or below. The mechanism which causes the slimy material to appear acts as though it is a micellar system with an isoelectric point between pH 6 and 7.

A series of detailed chemical analyses were conducted on the solids formed in the parent liquor, the retentate and the concentrate as well as the solids flushed out of the pilot unit during the washing operation.

One of the samples most fully investigated was Sample #3, January 19, 1973, which was a water flushing from the pilot plant. Details are given in Appendix E.

From such analyses, it appears that the basic slime forming material is polysaccharidic in nature and there is some component of long chain hydrocarbon materials. Materials derived from the cellulosic fibers are believed to be the basic contributors to this slime.

One other set of observations of merit in this discussion is that during ultrafiltration of both caustic extract filtrates and decker effluents with bulk pH of about 7, the concentrates displayed pH's of about 5-6 and the permeates had pH's 1-2 points higher.

From considerations such as these, it is hypothesized that the slime forming and membrane fouling constituents are present in the effluents predominantly as small micellar structures. These constituents are derived from the cellulose fibers and may be stabilized in the liquid by small amounts of tall oil soaps.

It is hypothesized further that the concentration effects at the membrane surface, and the concommittant pH depression, cause the micellar structure to agglomerate, resulting in the slime formation on the membrane surface.

The results obtained thus far in developing some understanding of this solid formation problem suggest that the formation of these materials, and membrane flux decline, can be minimized by:

--operating the system at as high a pH as feasible consistent with the use of cellulose acetate membranes;

--operating the membrane modules at high feed flow to minimize concentration polarization.

2. Plugging of Membrane Cartridge Flow Channels

The formation of slimes on the membrane surface can be greatly aggravated by blockage of the cartridge flow channels. Specifically, if the flow channels become partly blocked, flow channeling through only part of the cartridge results in a part of the membrane area being exposed to stagnant, or nearly stagnant, feed. Concentration polarization and slime formation become severe, and part of the membrane area becomes ineffective. Also, when the brine seals "flipped" flow bypassed the cartridges, at least in part, and the reduced net feed flow led to greater concentration polarization and membrane fouling.

The flow-channel plugging problem was encountered for all membrane cartridges with standard mesh spacers. The cartridges in shells 1a and 1b which operated at very high feed flow (∿7-10 gpm) showed substantially less collection of solids - as concluded from flux stability and nearly complete recovery of water flux on cleaning.

Other cartridges which became plugged did so irreversibly. Solids continued to accumulate with time, until the brine seals flipped. Autopsies of a few cartridges with high pressure drop showed extensive amounts of a grayish gelatinous material which filled the mesh spacer, and presumably, occluded membrane area.

For the TJ Engineering cartridges another problem arose. The brine seal was on the downstream end of the cartridge. Thus, the entire cartridge exterior was at the cartridge inlet pressure, while the interior pressure was at the inlet pressure, less the pressure drop to that point in the cartridge. If a 20 psig pressure differential existed across a cartridge (inlet to outlet), the static pressure difference between the exterior and interior, at the outlet end, would be 20 psig. This resulted in a net compressive force being exerted on the cartridges.

Since the TJ Engineering cartridges were loosely wound, extensive deformation occurred. This further aggravated the internal flow maldistribution problem.

Finally, in addition to flow channeling within a cartridge, channeling occurred among the Stage 1 shells. This appears to have been an unstable situation. When a flow maldistribution occurred, the shell with high flow remained clean, while the low flow shells continued to collect solids and plug since the low flow was not able to sweep the accumulated particulates out of the modules.

3. Prefiltration Efficiency

The efficiency of suspended solids removal greatly influenced the degree of membrane cartridge plugging by suspended solids, and hence, the membrane fouling characteristics as discussed above. In addition, depth and precoat filtration could have removed colloidal material which contributed, in part, to the slime formation.

4. Feed Circulation Rate

As discussed above, increasing the feed circulation rate reduced the flux decline for two reasons. First, it reduced the rate of particulate collection within the modules. Second, it reduced concentration polarization and, hence, the rate of slime formation.

F. CLEANING PROCEDURES AND EFFICIENCY

1. Procedures

The cleaning procedure that was found to be the most effective consisted of the following three steps:

Step 1: Low-pressure and high-flow water flush of the pilot plant in a once-through manner.
Step 2: Low-pressure and high-flow detergent cleaning for about 30 minutes with recirculation of the detergent solution.
Step 3: Repetition of Step 1 at the end of the detergent cleaning.

The water flushing was the most important part of the cleaning procedure and was done at 20-50 psig and 2-3 gpm per shell. Reverse-flow flushing was found to clean the system faster than forward-flow flushing.

Detergent cleaning was not always required and sometimes was performed on alternate days. However, on most occasions, detergent cleaning helped in removal of the gel-type foulant at a faster rate. After 12/5/72, the pilot plant was cleaned only at the end of every 15-22 hrs of continuous operation. The detergent cleaning part consisted of circulating about 40 gallons of cleaning solution, containing about 1% neutral or slightly alkaline detergent, for about 30 minutes.

It was found that either one of the following sets of operating conditions for detergent cleaning was effective:

Procedure used during December and January	10-40 psig pressure 2-3 gpm circulation rate per shell 100°F
Procedure used during October, February and March	40-70 psig pressure 4-5 gpm circulation rate per module 100°F

The following detergents were used for cleaning:

a) Ultraclean, Part A.; Abcor, Inc. (a phosphate type low-alkaline detergent).
b) Tide (pH adjusted to 7).
c) 1% EDTA + 1% Triton X-100 (Rohm & Haas Co.).
d) 1% citric acid + 1% Tergitol 15-S-7 (Union Carbide).
e) Ultraclean (enzyme detergent, Abcor, Inc.).

It was found that cleaning efficiency was not noticeably dependent on detergent type. Ultraclean Part A, which does not require pH adjustment, was used most frequently. Tide was also used on several occasions.

2. Effect of Feed Prefiltration on Cleaning

Table 15 summarizes the effect of feed prefiltration and different cleaning procedures on pilot plant performance

TABLE 15

EFFECT OF FEED PREFILTRATION ON CLEANING

Operating Period	Feed Filtration Systems	Operating Time, hrs	Cleaning Procedures		Cleaning Efficiency
			Water Flushing	Detergent Cleaning	
8-19-72 to 9-26-72	Broughton 10μ Filter	4-10	Inadequate; needed modifications in the pipelines	Yes	Poor. Required about 4 hrs (2-3 times repetition of cleaning cycles) to recover flux.
9-27-72 to 10-5-72	a. Hydromation, depth filter and b. 1μ Cuno cartridge filter	20	"	Yes	Improved. Required about 2 hrs to clean the system.
10-17-72 to 11-27-72	a. Plate & Frame Press with wood flour precoat & body feed; and b. 1μ Cuno cartridge filter	20	"	Yes	Required about 2-2½ hrs to clean the system.
11-28-73 to 2-2-73	"	20	Modified piping improved flushing operation. See details in text for operating conditions.		Required about 1-1½ hrs to clean the system.
2-11-73 to 4-1-73	a. Sparkler leaf filter with wood flour precoat & body feed; and b. 1μ Cuno cartridge filters	20	"		Required about 1 hr to clean the system.

and cleaning efficiency. The pilot plant ultrafiltration rate and cleaning efficiency were very poor at startup of the plant (8/19/72 to 8/30/72), at which time the filtration system was grossly inadequate. By contrast, the pilot plant could be cleaned within an hour after the installation of efficient filtration and flushing systems.

3. Effect of Intermittent Shutdown on Ultrafiltration Rate

During a study with the single cartridge it was observed that substantial flux recovery was possible with a brief shutdown of the unit. Figure 43 shows the effect of shutdown on this recovery. However, during the pilot plant operation similar flux recovery was not obtainable. The probable reason for this is that, in the single cartridge case, the particulates and membrane foulants were released during shutdown and on restarting were flushed out of the system. There was negligible recirculation of foulants in the single cartridge case. However, during the pilot plant operation, foulants released during shutdown were picked up again either by the same membrane stage or a subsequent one because of the high degree of internal recirculation. This was the major reason for poor cleaning efficiency of the pilot plant during the period when it was not possible to flush on a once-through basis.

Intermittent shutdown of the system during cleaning was also found to improve the cleaning operation. The same held for flow interruption during the water flush cycle.

4. Cleaning Efficiency in Different Membrane Stages

The cleaning efficiency of the membrane cartridges is, for very obvious reasons, dependent on the flow distribution within the cartridges. If the brine seal of a cartridge fails and creates a short circuit, flow through the cartridge will be reduced, and hence the effectiveness of a flush or cleaning cycle. Also, if the flow distribution within the different parts of the brine channel is not uniform, those parts where there is little flow will remain fouled. Many of the TJ Engineering cartridges became deformed under the compressive force discussed above, thereby affecting the flow distribution within their brine channels. These cartridges, compared in particular to the Gulf cartridges, showed high pressure drop and inadequate cleaning efficiency as measured by water and effluent fluxes.

Membrane cartridges with poor ultrafiltration performance were not readily cleaned. Both characteristics can be traced to unsatisfactory flow distribution within the cartridges.

Stage 1 membrane modules were arranged in three parallel shells. The cleaning efficiency and the ultrafiltration rate of these shells were dependent on flow distribution among the shells, in addition to the flow distribution within each cartridge. The variation in flow distribution in the three shells depended upon the type and history of individual cartridges and their varying resistance, as discussed before. Figure 52 shows the effect of cleaning on Stage 1b. It is seen that cleaning was reasonably effective, and that the ultrafiltration rate remained relatively high. During the same period, Stages 1a and 1c however showed lower flux. These cartridges were inspected and found to have undergone severe deformation (compression). They had, as a result, low flow during cleaning and ineffective cleaning.

The three wide channel corrugated spacer cartridges installed in Stage 2 were cleaned most easily.

Figure 53 shows the effect of cleaning on the Stage 3 membrane cartridges. These cartridges had the longest operating life, from 11/1/72 to 4/1/73. Figure 53 shows that the water flux of these cartridges decreased with time. The main reason for this decline appears to be membrane compaction. The flux decline parameter, measured by the slope of a log-log plot of water flux vs. time was about -0.078 (Figure 54).

G. MODULE MECHANICAL FAILURES

During the operation of the pilot plant a variety of failures with several of the 45 TJ Engineering cartridges used occurred. Mechanical problems with the three Eastman and four Gulf cartridges were negligible. Appendix D details the different problems experienced during the pilot plant operation. By and large, these problems appeared to be four fold:

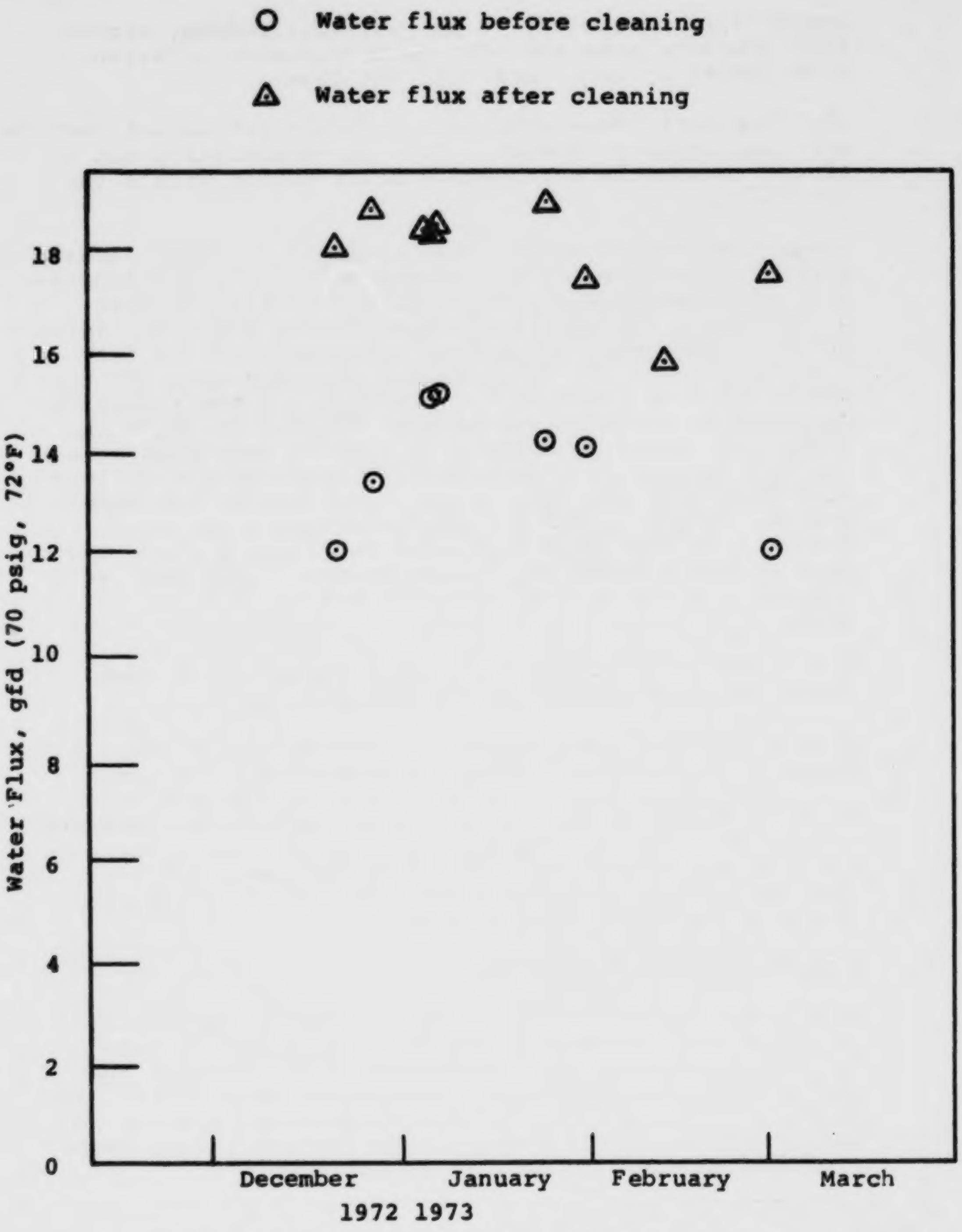


FIGURE 52: CLEANING EFFICIENCY OF STAGE 1b MEMBRANES

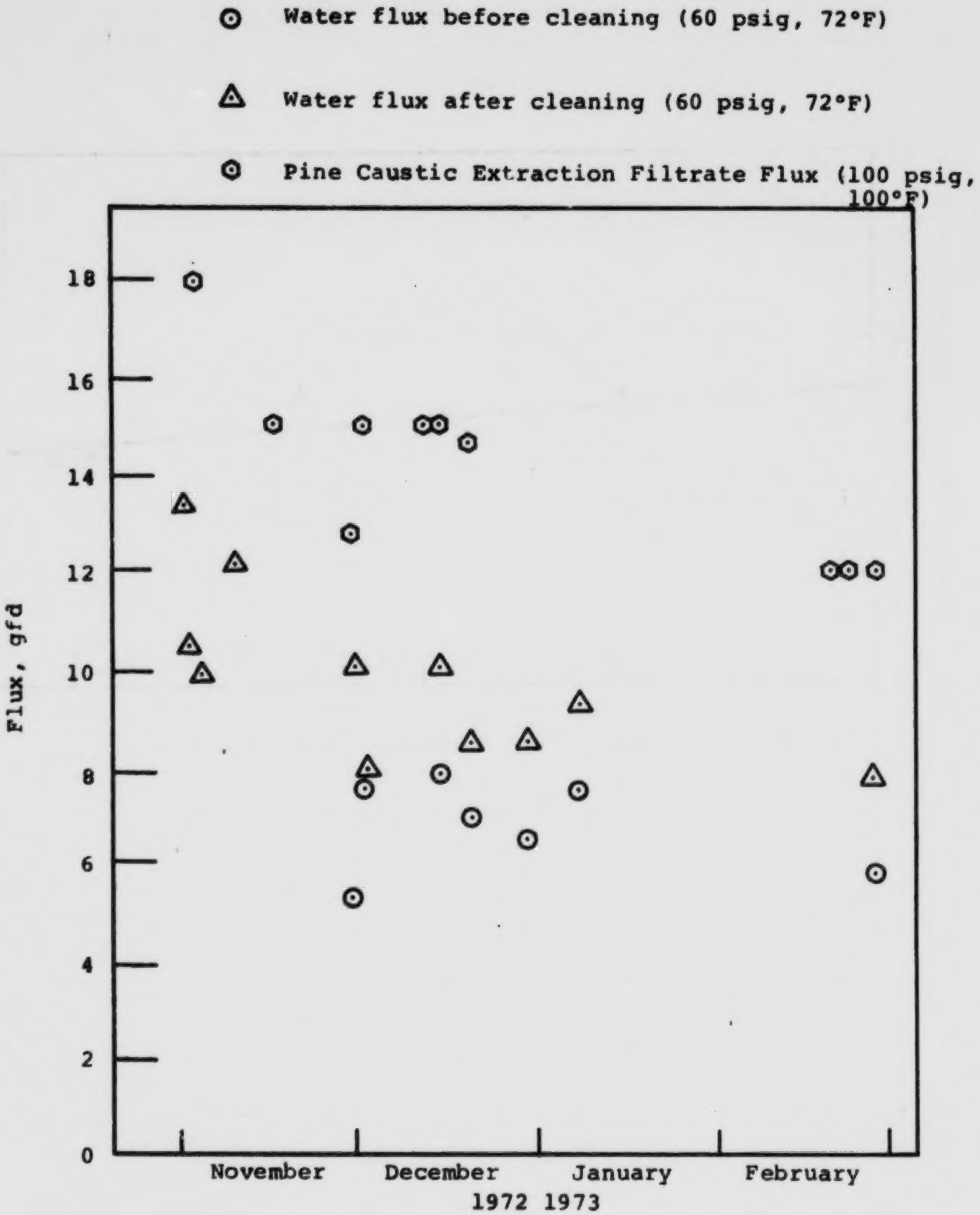


FIGURE 53: CLEANING EFFICIENCY OF STAGE 3 MEMBRANES

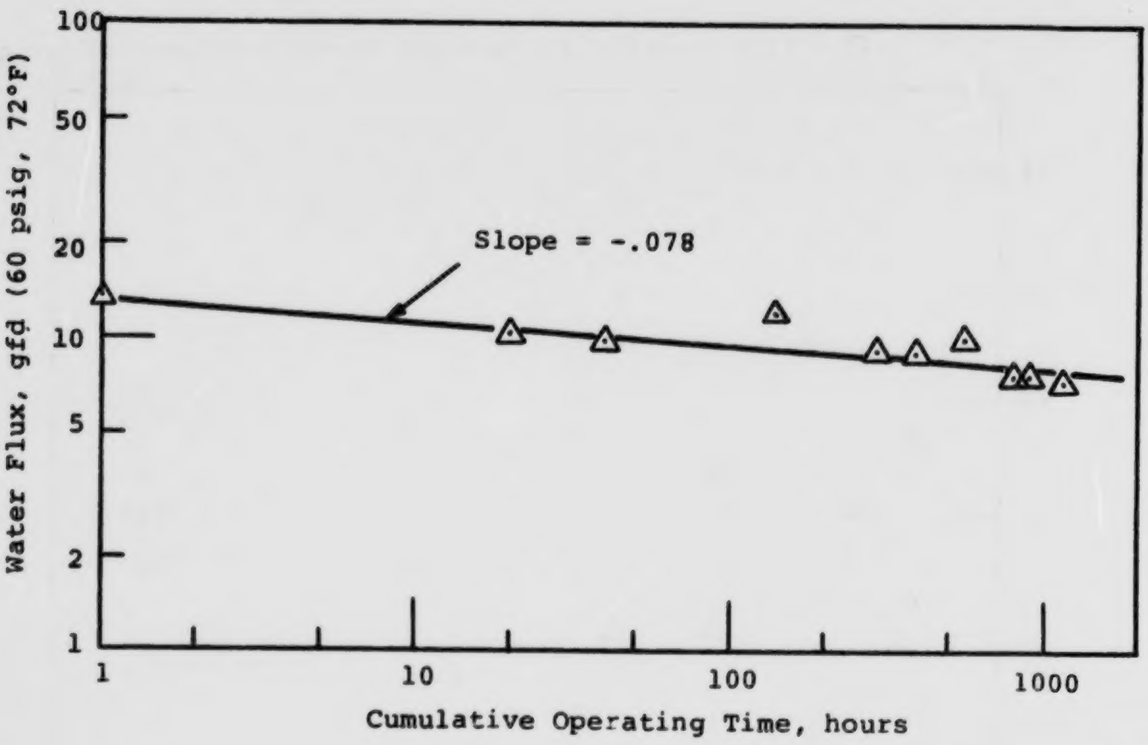


FIGURE 54: STAGE 3 COMPACTION CURVE

--O-ring failures;
--membrane cartridge compression, resulting in deformation of the cartridges;
--brine seal failures; and
--glue seam or membrane failures.

1. O-ring Failures. Membrane leaks in most cases were due to inadequate O-ring seals on the permeate collection tubes, and poor color rejection was corrected simply by replacing the O-ring seals. On at least five different dates involving stages 1a, 1c, 4 and 5, the poor color rejections were definitely identified as O-ring failures.

2. Membrane Cartridge Compression. TJ Engineering cartridges have brine seals on the downstream end, thus the exterior pressure on the cartridges is higher than the interior pressure due to pressure drop of feed flowing through the cartridge. Several cartridges were observed to "deform" and "shrink" from their round shape. These cartridges were primarily in stages where severe pressure drop across the cartridges was present.

It is suspected that module compression created dead-ends which could not be cleaned, thus resulting in low ultrafiltration rate and accumulation of fouling materials. The latter phenomenon created additional pressure drop, consequently more compression, and further aggravated the problem.

Gulf cartridges, however, did not show this problem. Gulf cartridges have brine seals on the upstream end, and the pressure within the cartridge is higher than the exterior pressure. The net result is that the Gulf cartridges received an "expansive" force, which maintained the brine channel width intact and prevented dead-ends.

3. Brine Seal Failures. As the membrane cartridges collected solids and/or underwent compression, pressure drop across the cartridges increased. This resulted in brine seal reversal, permitting feed flow to bypass the membrane cartridges. It is possible that this effect accounted for gradually declining flux in several of the membrane shells.

The four Gulf cartridges which were in the system for a period of about seven months did not evidence brine seal reversal. At the end of the test period, however, three of the four brine seals were observed to have weak tapes which needed reinforcement. Several TJ Engineering cartridges required replacement of brine seals (mainly foam gaskets and tapes).

The outer wraps of almost all the cartridges remained in good shape despite the compression or expansion forces (10 to 20 psi per cartridge).

The pressure drop data show that the brine seals of the spiral cartridges probably failed to maintain a proper seal when the pressure drop across the seals reached about 20 psi.

4. Glue Seam or Membrane Failures. In some instances, leaks can only be attributed to membrane cartridge failures. There have been five failures of this kind involving stages 1b, 1c, 4 and 5. All cartridges were from TJ Engineering Co.

Membrane cartridge failures may have happened because of

- --leakage at the glue seam;
- --leakage at the material fold adjacent to the permeate collection tube;
- --membrane and/or backing material wrinkles causing direct leakage;
- --pinholes in membrane.

One membrane cartridge with poor color rejection was autopsied and was found to have glue seam failures in several places, especially at points where longitudinal wrinkling was present. The Gulf cartridges had membranes directly cast on membrane-support sheets. Gulf has reported that spiral modules made in this manner show more uniform brine channel thickness (28).

H. INCINERATOR STUDIES

Disposal of the concentrate from the ultrafiltration of pulp mill effluents is a problem only if the concentrate contains substantial amounts of chlorides. In the studies discussed here it was found that disposal of the concentrate from the pine caustic extraction filtrate does require special processing, but that the concentrate from the decker effluents can be beneficially used in the weak black liquor system. The following discussion applied, then, only to the concentrate from the pine caustic extraction filtrate.

The concentrate produced in ultrafiltration of the caustic extraction filtrate would contain about 20% total solids. These solids would be predominately chlorinated lignins with some amount of sodium salts such as sodium chloride.

The content of organic and inorganic chlorides in the concentrate makes disposition a problem if it is to be injected into any of the present pulp mill streams. For example, if the concentrate were processed in the black liquor recovery system, about 500 pounds per day of hydrogen chloride would be liberated in the recovery boiler from combustion of the chlorinated lignins and also about 500 pounds per day of sodium chloride would be introduced into the recovery boiler from the inorganic solids in the concentrate. These chloride-containing materials could lead to corrosion problems in the boilers, dusting problems in the boiler stacks due to the liberation of finely divided sodium chloride, and also to raising the chloride level in the pulping system, as a function of the material recycle system.

Injecting the concentrate into the lime mud kilns would lead to similar problems.

As a result of the engineering evaluations it was concluded that the disposition of the concentrate from the pine caustic extraction filtrate would require either an incinerator specifically designed to burn organic chlorides, or, evaporation to high solids and subsequent admixture with primary sludge for disposal as land fill.

Laboratory studies have been conducted on evaporation of concentrate to various levels of solids. In all of the tests the material dried with no apparent increase in viscosity to about 80% solids. The material does not scale nor foam when concentrated by evaporation. When a 50% solids concentrate was mixed with primary sludge, the admixed sludge was judged to be satisfactory for land fill. As a result of the laboratory tests it was concluded that evaporation of the concentrate to about 50% solids could be done in commercially available equipment using waste hydrogen available on the plant site as the heat source. The 50% concentrate would be added to the dewatered primary sludge and carried to land fill. This method of disposal would add 10% to the present daily primary sludge weight.

Concentrate from the ultrafiltration unit was incinerated in February at the John Zink Company, Tulsa, Oklahoma pilot facility. The purpose of the tests was to establish parameters on which to base a budgetary estimate for a full scale incineration system to dispose of the concentrate.

The tests demonstrated that the Zink incinerator will provide a method of disposing of the concentrate.

The operation of the incinerator requires the addition of supplementary fuel. To minimize operating costs, hydrogen from the electrochemical cells, which is presently unused, would be burned.

The tests demonstrated the need for the use of a venturi scrubber and a vent scrubber to dispose of the potentially plume-forming, finely-divided inorganic salts formed in the combustion of the concentrate. In a full scale installation, the pine caustic extraction filtrate, prior to neutralization, would be used as the scrubbing fluid for the venturi scrubber and the stack scrubber. The partially neutralized pine caustic extraction filtrate would then be treated in the ultrafiltration system.

The economic feasibility of the use of an incinerator for the disposal of the concentrate is heavily dependent on the availability of an inexpensive fuel source. If hydrogen is used as the fuel source, the fuel costs will be those associated with the capital and operating costs of the system required to deliver it to the incinerator.

The partial evaporation system for disposal of the concentrate requires less energy input but does require increased costs for land fill operations with the primary sludge.

The capital and operating costs of both systems are improved, the more concentrated the solids level produced by the ultrafiltration system. These costs are also strongly influenced by the salt rejection characteristics of the specific membrane system used; the incinerator system is more cost sensitive than the evaporation system. If low salt rejection membrane systems are used, the ratio of the organic content to inorganic content in the concentrate will be increased and for a given solids level the heating value of the concentrate will be increased. Increasing the organic solids content of the concentrate will yield a material of both higher heating value per unit concentrate weight and also less water to be evaporated in the combustor.

The choice of the system for disposal of the concentrate depends on several design parameters of an ultrafiltration plant as well as the specific plant site for which it is designed. At the present there are at least two technically feasible methods for disposing of the ultrafiltration concentrate from pine caustic extraction filtrate. In the capital estimates for these systems (discussed in Section VI.A.) compromise cost figures are used.

SECTION VI

FULL SCALE PLANT DESIGN AND COSTS

A. FIRST STAGE PINE CAUSTIC EXTRACTION FILTRATE SYSTEM

1. Flow Schematic

A general flow schematic has been presented in the Introduction as Figure 2. A second generalized process flow schematic, more specific for the ultrafiltration system, is given in Figure 55. First-stage pine caustic extraction filtrate flows from the pine pulp bleachery through a pretreatment system. Pretreatment consists of neutralization and filtration. The treated feed is cooled to 100°F. Although future membranes would process pine caustic extraction filtrate at its normal temperature (120-130°F), current membranes probably cannot withstand a temperature in excess of 100°F and exhibit long life. It is to be noted that no long-term life data are available at temperatures higher than 100°F for membrane systems treating pine caustic extraction filtrate.

The pretreated and cooled feed is concentrated in the ultrafiltration system. The permeate (treated effluent) is sewered and flows to the mill's waste treatment system. The concentrate from the ultrafiltration system is used to sluice the filter cake, and this suspension is pumped to an evaporator and evaporated to 50% solids. Excess hydrogen currently available in the mill from the electrochemical cells will be used as fuel. The evaporator discharge will be mixed with primary sludge and disposed of as land fill.

A more detailed process flow schematic is given in Figures 56 and 57. These two figures differ in that Figure 56 is for a low-flow membrane system (spiral wound cartridges with standard mesh spacers), and Figure 57 is for high-flow membranes (spiral wound cartridges with corrugated spacers). The reasons for examining these two cases are discussed below.

Sulfuric acid is mixed with pine caustic extraction

Preceding page blank

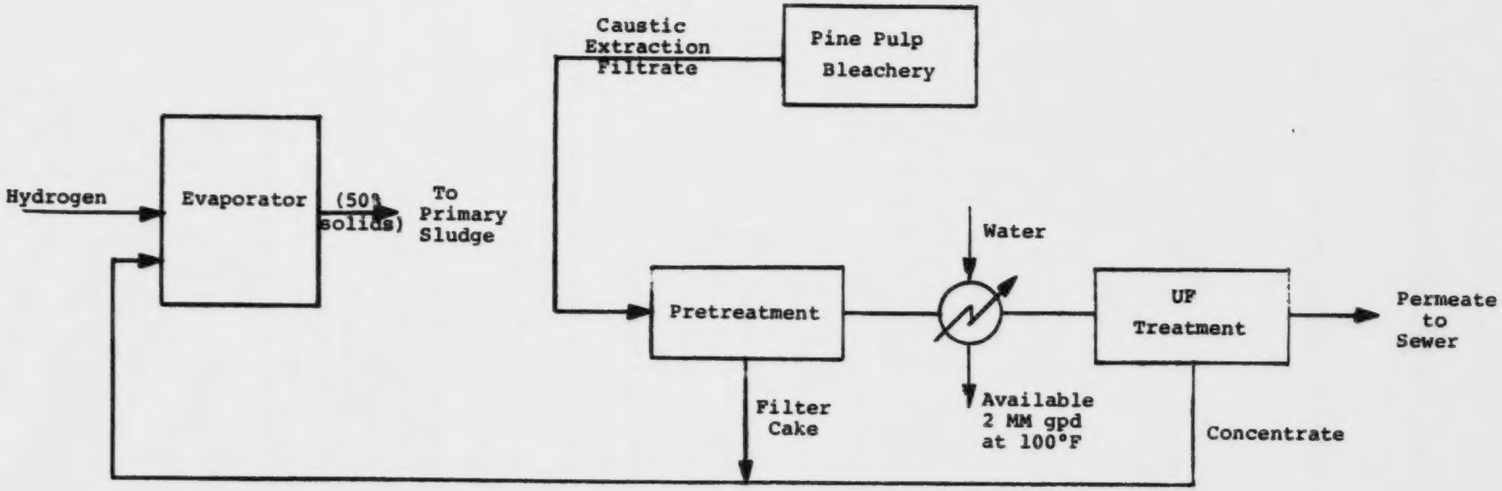

Caustic
Extraction
Filtrate
Pine Pulp
Bleachery
Hydrogen
Evaporator
(50%
solids)
To
Primary
Sludge
Water
Pretreatment
UF
Treatment
Permeate
to
Sewer
Filter
Cake
Available
2 MM gpd
at 100°F
Concentrate

FIGURE 55: SIMPLIFIED FLOW SCHEMATIC: TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE

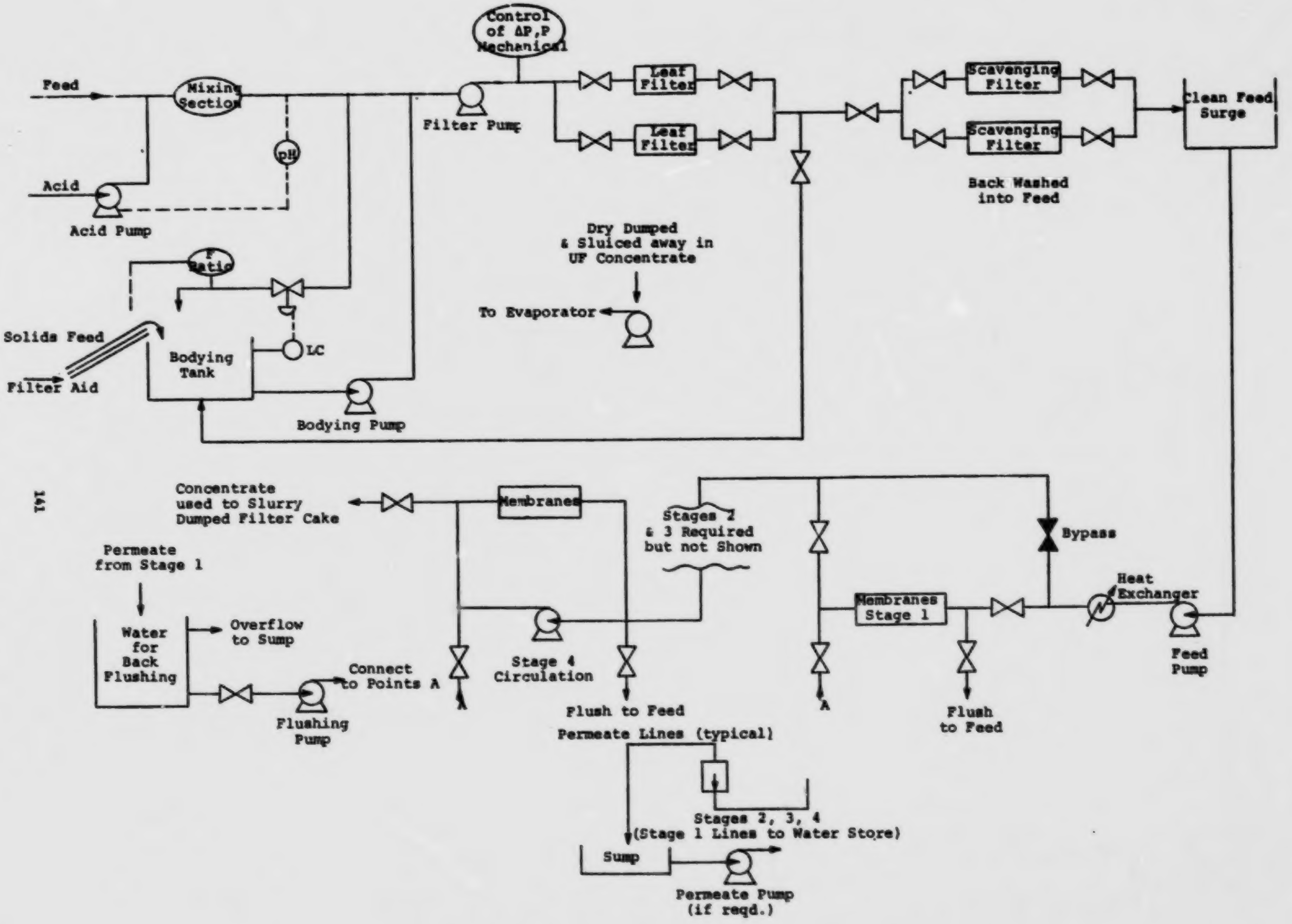

Feed
Mixing Section
pH
Acid
Acid Pump
Control of ΔP,P Mechanical
Filter Pump
Leaf Filter
Leaf Filter
Scavenging Filter
Scavenging Filter
Clean Feed Surge
Back Washed into Feed
F Ratio
Solids Feed
Filter Aid
Bodying Tank
LC
Bodying Pump
Dry Dumped & Sluiced away in UF Concentrate
To Evaporator
Concentrate used to Slurry Dumped Filter Cake
Membranes
Stages 2 & 3 Required but not Shown
Bypass
Heat Exchanger
Membranes Stage 1
Feed Pump
Permeate from Stage 1
Water for Back Flushing
Overflow to Sump
Flushing Pump
Connect to Points A
A
Stage 4 Circulation
A
Flush to Feed
Flush to Feed
Permeate Lines (typical)
Stages 2, 3, 4
(Stage 1 Lines to Water Store)
Sump
Permeate Pump (if reqd.)

FIGURE 56: FLOW SCHEMATIC FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE:

LOW FLOW CASE

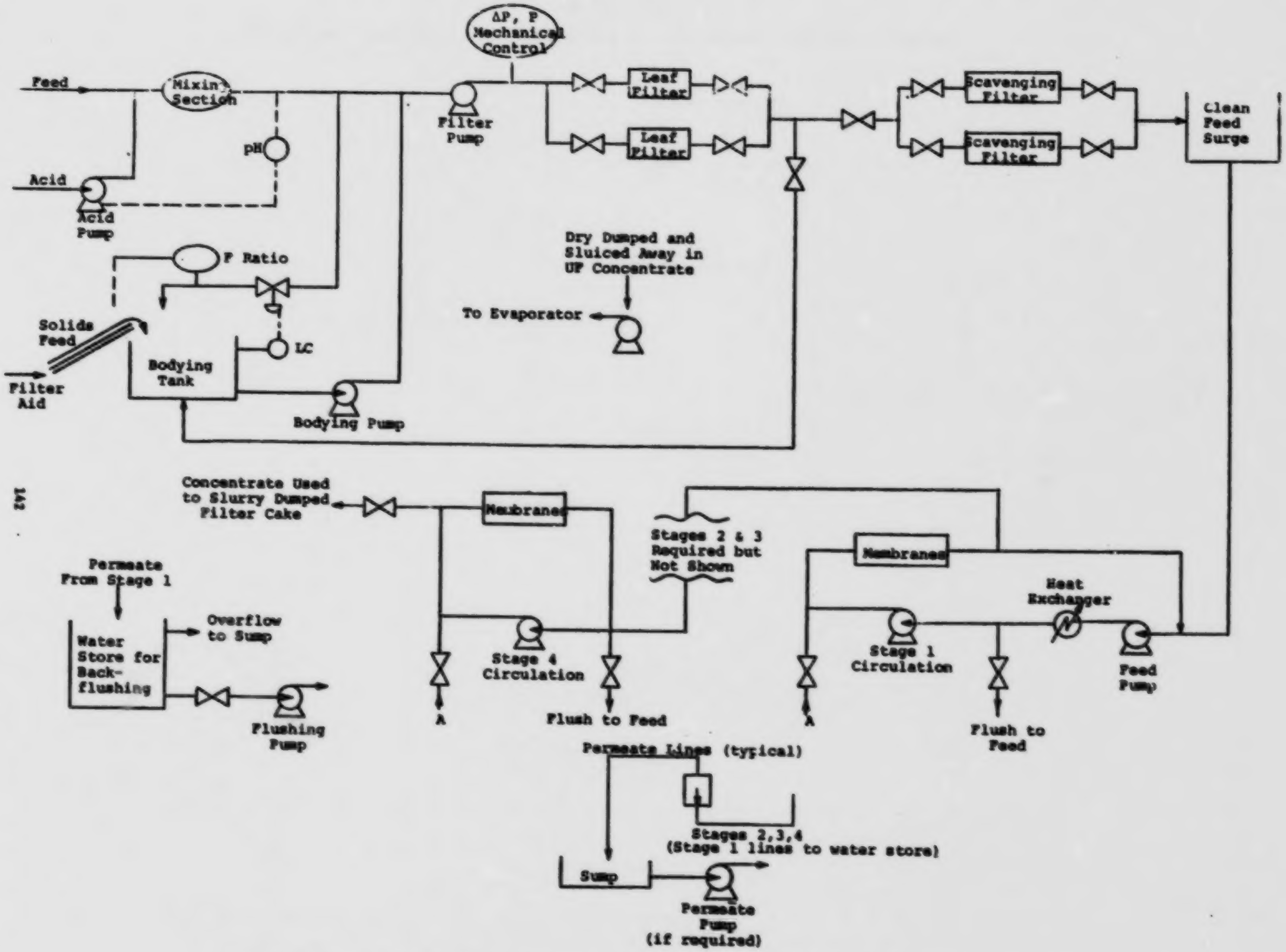

Feed
Mixing Section
ΔP, P Mechanical Control
Filter Pump
Leaf Filter
Leaf Filter
Scavenging Filter
Scavenging Filter
Clean Feed Surge
Acid
pH
Acid Pump
F Ratio
Solids Feed
Filter Aid
LC
Bodying Tank
Bodying Pump
Dry Dumped and Sluiced Away in UF Concentrate
To Evaporator
Concentrate Used to Slurry Dumped Filter Cake
Membranes
Stages 2 & 3 Required but Not Shown
Membranes
Heat Exchanger
Stage 4 Circulation
Stage 1 Circulation
Feed Pump
A
Flush to Feed
A
Flush to Feed
Permeate From Stage 1
Water Store for Back-flushing
Overflow to Sump
Flushing Pump
Permeate Lines (typical)
Stages 2,3,4 (Stage 1 lines to water store)
Sump
Permeate Pump (if required)

FIGURE 57: FLOW SCHEMATIC FOR TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE HIGH FLOW CASE

filtrate in an in-line mixing section. The rate of acid addition is controlled by a downstream pH probe and control system.

A small portion of the neutralized feed flows to a bodying tank. Filter aid (tentatively wood flour) is added by a solids feeder. The rate of addition of filter aid is such as to maintain a body feed level of 50-100 ppm. The body feed slurry is pumped with the bodying pump back into the feed line, where it mixes with the neutralized pine caustic extract. This stream is pumped through leaf filters to remove most of the suspended solids contained in the feed. Instrumentation for the filter system will control operating pressure, pressure drop, and mechanical operation. Dry cake will be dumped, sluiced away with the ultrafiltration concentrate, and sent to the evaporator.

A bypass line to the bodying tank on the downstream side of the leaf filters is used to apply the precoat. Specifically, after a cake has been dumped, pine caustic extraction filtrate, with filter aid added, is circulated through the leaf filters and returned to the bodying tank. When an adequate precoat has built up, normal flow into the membrane system is resumed.

The feed from the leaf filters is further treated in backwashable scavenging filters. These polishing filters remove any filter aid or other large particles which pass through the leaf filters. The solids' discharge from the scavenger filters (backwash) is mixed with the feed pine caustic extraction filtrate for additional solids removal in the leaf filters.

The filtered, neutralized feed flows to a surge tank prior to treatment in the ultrafiltration system. This surge capacity allows for short-term interruption in the feed flow or filter operation, without causing an immediate membrane system shutdown. Feed from the surge tank is pumped through a heat exchanger and cooled to 100°F. Flow is then through a series of four membrane stages. For the low-flow membranes, Stage 1 can be operated on a once-through basis; that is, it is not necessary to recirculate concentrate within the Stage 1 flow loop. The concentrate from Stage 4 is used to sluice the filter cake, and flows to the evaporator for further concentration. The permeate from Stage 1, used for backflushing the membranes, flows to a water storage tank. The permeates from Stages 2, 3, and 4 are collected in a sump, and pumped to the sewer by a permeate pump. The membrane stages will be set up for reverse-flow flushing, an operation which has been found to be effective in membrane cleanup. The Stage 1 permeate will be used for this purpose. In addition, if detergent cleaning of the membranes is required, the Stage 1 permeate storage tank will be used to mix the detergent solution.

The high-flow membrane system shown in Figure 57 is identical to the low-flow system, except that recirculation in Stage 1 is required to maintain high velocity through the membranes.

Details of the equipment for these process flow schematics are given below with cost estimates.

2. Equipment Description and Capital Cost Estimates

Five capital cost estimates for full-scale, battery-limits plants are presented. These cases have been prepared to provide some insight into the relative cost sensitivity of the various parts of the process system. The significant process parameters used for each case are shown in Table 16.

The equipment cost estimates presented are based on budgetary estimates obtained from potential vendors. In the following, Case 1 will be described in detail; the other cases will be presented as modifications of Case 1.

Membrane Module Alternatives--Different Flow Channel Spacers. Spiral wound membranes will be used. These membrane cartridges are currently available from Gulf Environmental Systems with either the standard mesh spacers (60 ft^2/cartridge @ \$1.50/ft^2) or corrugated spacers (40 ft^2/cartridge @ \$2.25/ft^2). In the pilot plant operation, as described above, most of the membrane cartridges had the standard mesh spacers. In addition to the mechanical failures encountered with the T. J. Engineering cartridges, all cartridges with mesh spacers were difficult to clean. In the early part of the pilot plant operation, an inability to thoroughly clean the membrane cartridges resulted in increased pressure drop across the cartridges with time, as well as reduced capacity. It was subsequently ob-

TABLE 16

CASES FOR CAPITAL COST ESTIMATES
PINE CAUSTIC EXTRACTION FILTRATE

Case	Pine Caustic Extraction Filtrate Flow $(\times 10^6$ gpd)	Membrane Modules Low Flow (mesh spacer)	Membrane Modules High Flow (corrugated spacer)	Membrane Flux, gal/day-ft^2	Circulation Rate gpm/membrane cartridge	Membrane Cost \$/sq ft	Prefiltration Precoat Filter	Prefiltration Backwashable Depth Filter	Prefiltration Screen Filter	Operating Manpower, No. of Men
1	2	x		18	8	$1.50	x			5
2	3		x	18	15	$2.25	x			5
3	2		x	18	15	$2.25			x	4
4	2	x		18	8	$1.50		x		4
5	1	x		18	8	$1.50		x		4

served that improved prefiltration and strict adherence to prescribed cleaning procedures alleviated the problems associated with mesh-spacer cartridges. That is, a "steady state" operation was achieved. Specifically, over long periods of operation it was possible to return repeatedly to a base-line membrane flux and cartridge pressure drop after cleanup.

Experiments with membrane cartridges with corrugated spacers suggest that it will be substantially easier to clean these cartridges, possibly by water flushing alone. In particular, reverse-flow flushing is anticipated to be substantially more effective for corrugated-spacer cartridges that for mesh-spacer cartridges. Thus, corrugated-spacer cartridges offer several possible advantages. These are:

--maintenance of a higher average flux (reduced membrane area requirement);
--facilitated cleanup, reducing down-time for cleanup, and possibly eliminating the need for detergent cleaning;
--longer membrane cartridge life; and
--possible operation without precoat filtration.

These advantages can be of substantial importance in terms of process costs. It is to be noted, however, that these factors have not been demonstrated to date.

The cost figures for the two different membrane cartridges given above are identical, i.e. $90/cartridge. These are the costs currently quoted by Gulf Environmental Systems. It is not clear why costs should be independent of the square footage of membrane in each cartridge. On the contrary, for large-scale production costs should be proportional more to membrane area, and not to the number of cartridges. For present estimates, however, the Gulf price structure will be used, although it will tend to give a conservative picture for corrugated-spacer membrane cartridges. The two cases are summarized below.

1. \$1.50/ft^2, for membrane cartridges with mesh spacers. A circulation rate through these cartridges of 8 gpm is assumed to be sufficient to minimize membrane fouling and pre-

vent extensive solids collection within the cartridges. These membranes will be assumed to have a three-year life. Precoat filtration is required.

2. $2.25/ft^2, for membrane cartridges with corrugated spacers. A circulation rate of 15 gpm through the membrane cartridges is assumed to be sufficient to maintain cleanliness and avoid cartridge plugging. Membrane cartridge life is assumed to be three years. Precoat filtration may not be required.

The relatively high flows through the membrane cartridges have been selected on the basis of the pilot plant operation. For shells (with mesh-spacer cartridges) which were operated at high feed flow, membrane fouling and particulate collection within the cartridges was greatly reduced. The three-year life assumed is an important, but not critical, factor. For example, a two-year membrane life would add about 4¢/1000 gal. to the plant operating costs (detailed below).

For design purposes, it has been assumed that corrugated-spacer cartridges can be obtained with 60 ft^2 of membrane area. The desirability of having a high square footage per cartridge relates to mechanical problems encountered in the pilot plant operation. Specifically, it is highly desirable to minimize the total number of seals, gaskets, and glue seams in the system since these are the points of potential operating problems.

The flow characteristics of the two different membrane cartridges are given in Table 17.

TABLE 17

CHARACTERISTICS OF MEMBRANE CARTRIDGES

Spacer	Mesh	Corrugated
Feed Pressure	120 psi	120 psi
Outlet Pressure	80-90 psi	80-90 psi
Circulation Rate	8 gpm	15 gpm
Pressure Drop	8 psi/cartridge	8 psi/cartridge
Maximum Number of Cartridges in Series	4	4

The operating pressures chosen are 120 psig at the inlet to the cartridges and 80-90 psig at the outlet from the cartridges. At the circulation rates given, pressure drop per cartridge will be about 8 psi. Correspondingly, the maximum number of cartridges which can be assembled in a series configuration is four. Thus, four cartridges will be installed in a single housing, and this will be denoted as a "membrane module".

In any single stage, capacity will be fixed by the total membrane area. Since more than four membrane cartridges will be required, it is necessary to pipe membrane modules in parallel. The number of parallel modules is determined by the total area requirement.

The costs of membrane modules for cartridges with mesh and corrugated spacers are given in Table 18.

TABLE 18

CHARACTERISTICS AND COSTS OF MEMBRANE MODULES

Spacers	Mesh	Corrugated
Circulation Rate	8 gpm	15 gpm
Area	240 ft^2	240 ft^2
Bare Membrane Cost	$360	$540
Housing and Installation	$390	$390
TOTAL COST	$750	$930

The modules with mesh spacers will cost $750 each; and those with corrugated spacers, $930 each. The cost difference is due solely to the greater bare membrane area cost for corrugated-spacer spirals.

Installation includes all racks, internal piping, shipping, and on-site direct labor. Excluded are costs for pumps, the building and foundations, engineering design, materials procurement, construction supervision, and startup.

Membrane cartridges with corrugated spacers will probably be somewhat larger physically than membrane cartridges with mesh spacers, and the housings could cost more. However, for present purposes, the housing and installation costs have been assumed to be the same.

a. Details of Case 1 Design

(i) Design Bases. The Case 1 design bases are as follows:

- --2 x 10^6 gpd pine caustic extraction filtrate processed;
- --mesh-spacer membrane cartridges used;
- --precoat filtration needed; and
- --membrane flux of 18 gal./day-ft^2.

(ii) Feed Rate and Its Effect on Plant Size. It has been assumed that the pine caustic extraction filtrate flow is constant at 2 x 10^6 gpd (1,390 gal./min). However, membrane flux depends on membrane cleanliness and changes with time. In any case, the plant has been sized for a flux of 18 gal./day-ft^2, which is an average flux over the operating period between cleaning cycles. Flux varies from 25-30 gal./day-ft^2 for clean membranes to 10-15 gal./day-ft^2 for fouled membranes.

The simplest way to operate a system is to process feed at a rate greater than 2 x 10^6 gpd immediately after membrane cleaning, and then to reduce the feed flow into the membrane plant as the membranes become fouled. During the latter part of this operation it would be necessary to accumulate surplus feed for use during the next run immediately after cleaning. However this requires supplying a large surge capacity for the feed, which would be prohibitively expensive. Therefore, operation will be such that a small fraction (one-quarter or less) of the membrane plant will be flushed and cleaned at any single time. That is, the flushing/cleaning sequence will be cycled through isolatable sections of the ultrafiltration plant. This will increase piping and operating costs somewhat, but a net benefit will accrue due to the elimination of a requirement for a large feed surge capacity.

It is further assumed that each segment of the plant can be cleaned in one hour per day. Consequently, the operating time available is 23 hrs/day.

Excess membrane area will be required to produce the permeate used for membrane flushing. Since the plant volume is about 10,000 gals., conservatively not more than 100,000 gpd will be required for once-a-day flushing and cleaning. This will increase the membrane plant area requirement (capacity) by 5% or less.

Finally, some surge capacity must be available, and a one-hour surge (80,000 gals.) will be provided.

(iii) Plant Size. The ultrafiltration system must process 2.1 x 10^6 gals. in 23 hrs, at an average flux of 18 gal./day-ft^2. The permeate rate is essentially the same, since more than 98.5% of the feed must pass through the membranes. Thus, the calculation of the membrane area required is:

$$\text{Membrane area} = 2.1 \times 10^6 \times 24/23 \times \frac{1}{18} = 122{,}000 \text{ ft}^2.$$

The minimum size of the pretreatment section of the process, including the filters, is 2.1 x 10^6 gals. within 23 hrs, or 1,500 gpm. A slightly larger filtration system will be chosen to allow for cleaning the filtration section and emergencies. Specifically, the filtration section will be designed to handle 2.1 x 10^6 gals. within 22 hrs, or 1,600 gpm.

(iv) Neutralization and Filtration Sections. The feed will be neutralized with sulfuric acid before filtering. Based on pilot plant experience, the feed will be filtered first in a precoat filter, adding filter aid to body the liquid at 50-100 ppm. This will be followed by a scavenging filter. The filter aid tentatively will be wood flour. Costs for equipment in the neutralization and filtration sections are given below.

	Cost
Acid store - 500 gals., assumed to be available.	n/c
Acid pump and piping - The probable acid use rate is 8000 lb/day, about 0.4 gpm. On-off control through a low pressure diaphragm metering pump (∿1/2 hp) and iron piping.	$2,000

	Cost
pH measurement and control - In line pH electrodes with a recorder, on-off control and alarm switches, installed.	$6,000
Filter aid solids handling - Filter aid will be received in bags. Daily usage about 1,000 lb/day (about 70 ft^3/day). Hopper and controlled rate conveyor.	$3,000
Bodying tank - 600 gal. carbon steel. 5 ft diameter x 4 ft high, with 1/4 hp agitator.	$1,600
Bodying pump - 1 gpm, 1/2 hp.	$ 500
Precoat filter - Tests at Sparkler Manufacturing indicate that an achievable filtration rate is 1.5 gpm/ft^2. For conservative design purposes, 1 gpm/ft^2 is assumed and 1,600 ft^2 filter area will be installed. Two units, Sparkler MCRO-1000-3, each having 1000 ft^2, vertical leaf, horizontal tank filters for dry case discharge cost $35,100. Scaled to 1,600 ft^2.	$30,000
Piping and valving - for the precoat filters.	$ 7,000
Scavenging filters - 10 units Velmac backflushable felt-type filters, each 13" diameter and 62" high, rated 100-200 gpm.	$10,000
Piping and valving - for scavenging filters.	$10,000
In-line mixing section -	$ 1,500
Instruments and controls - other than for pH, for measuring pressure and differential pressure and automating filter flushing.	$16,000
Filter pump - Size 8 x 10-13 to pump 1,600 gpm into 94 feet (40 psi), 60 hp. Operated at 62% efficiency. In iron. Driven and started, not installed or connected.	$ 3,200

	Cost
Back washing and other filter cake removal - The scavenging filters will be back washed into the feed. The precoat filters will be dumped (dry), probably 2 or 3 times each day. About 100 ft^3/day of cake will be discharged. This cake will be mixed with 20,000-30,000 gpd of concentrate and conveyed either to the evaporator (for pine caustic extraction filtrate) or to the black liquor plant (for decker effluent).	$ 800
Slurry pump - to remove the mixture of filter cake and concentrate, 14 gpm.	$ 400
SUBTOTAL FOR EQUIPMENT	$85,000
Transportation, based on non-instrument, heavy pieces, totalling about $63,000 and coming from 800 to 1,000 miles.	$ 2,500
Installation, direct labor only, including rigging, pipe fitting and electrical connections of (approx): 2 pieces filter hydraulic motors, 1/2 hp acid pump, 1/2 hp dry feeder, 1/2 hp tank stirrer, 1/4 hp filter pump, 60 hp instruments and controls, 20 amp max. filter cake mixer, 1/2 hp slurry pump, 1 hp	$12,600
TOTAL PRETREATMENT SECTION (Engineering design, equipment procurement, supervision and startup not included)	$100,100

(v) Ultrafiltration Section. 122,000 ft^2 of membrane area will be used. This will be incorporated into four stages (Figure 56); details are given in Table 19.

Each membrane module requires 8 gpm inlet feed flow. Two-hundred parallel modules have been provided in

TABLE 19

ULTRAFILTRATION SECTION DESIGN DETAILS AND COSTS--CASE 1

Stage Number	1	2	3	4
Number of Modules	200	160	90	60
Circulation Rates (gpm)	1600 (feed)	1280	720	480
Pump Differential (psi)	120 (feed)	40	40	40
Utilized Horse Power	175	50	25	20
Efficiency for Estimating h.p.	64%	60%	68%	56%
Approximate Pump Size	6x8-16	6x8-13	6x8-11	4x6
Cost Estimate (Driven, Started and Installed Electrical Connection	$5,200	$3,000	$2,600	$1,400

Additional Equipment

Flush Pump:	1600 gpm, 40 psi, 60 HP	$ 3,200.
Permeate Pump:	same as Flush Pump	$ 3,200.
Water Store:	20,000 gallons, delivered and installed	$10,000.
Flush and drain piping, installed		$ 9,000.

Stage 1, requiring 1,600 gpm. The feed rate of 1,520 gpm is adequate for once-through operation and circulation in Stage 1 is not required. The feed pump delivers 120 psig, and a Stage 1 circulation pump is not provided.

The feed to Stage 2 is 920 gpm, which can feed 120 parallel membrane modules on a once-through basis. Since a greater number of modules is used, recirculation is necessary. Perhaps of greater importance is the difficulty of operating two, once-through stages in series. Specifically, an imbalance would occur since the second pump would upset the flow and pressures in the first stage.

Stages 3 and 4 would require circulation in any event since the net feed flow to each of the stage will be relatively low.

(vi) Summary of Ultrafiltration Section Costs.

	Cost
Membranes (510 x $750) (includes $183,600 for membrane cartridges)	$382,500
Pumps	18,600
Tanks and piping	19,000
Electrical connections for a total of 350 hp to 6 pumps (transformer excluded)	9,000
TOTAL (Engineering design, equipment procurement, supervision of construction, and startup not included)	$429,100

(vii) Other Equipment. Other equipment costs are given below.

	Cost
Clean feed surge tank - will have about one hour's surge capacity (80,000 gals.), installed.	$ 30,000
Heat exchanger (counter current) - to cool 1,600 gpm from 130°F to 100°F using 1,600 gpm of water which is heated from 70°F to 100°F. Installed cost.	

	Cost
24 million BTU/hr 150 BTU/(hr)(ft^2)(°F) 30°F ΔT 5,350 ft^2	$ 75,000
Control panel -	$ 10,000
Building - about 40 ft x 50 ft concrete pad with sumps and pump pads of cast concrete. A prefabricated type of building 30 ft to cross beams. Very limited heating as the system releases substantial heat (electrical substation, if required, is not included).	$ 48,000
Evaporator - based on direct contact between flame and solution to concentrate 20,000-30,000 gpd of 20% solids to 50% solids. Assumed to be fueled by burning hydrogen available from Electrochemical Plant. The 50% solids stream will be disposed of with the primary sludge from the waste treatment plant. Incineration is preferable to evaporation and may be possible. However, preliminary capital costs provided by the John Zink Company are high compared to previous estimates. Furthermore, poor burning efficiency will require additional fuel consumption. Therefore, for present purposes, incineration is questionable. Evaporator, installed, including hydrogen handling system.	$125,000
Total Other Equipment -	$288,000
Engineering charges - including all design and drafting, equipment procurement, construction supervision, and shift engineers for startup and supervision for the first twelve months of operation.	$300,000

(viii) Capital Cost Summary, Case 1.

	Cost
Filtration and neutralization section	$100,000
Ultrafiltration section (includes membranes @$183,000)	$429,000
Other equipment and building	$288,000
Design, Administrative, and Supervision	$300,000
	$1,117,200
Contingency, @ 10%	111,700
TOTAL	$1,228,900

Total utilized energy is 374 hp excluding the flushing pump which is run only when a circulation pump is stopped.

b. Details of Case 2 Design

(i) Design Bases. The design bases for Case 2 are given below:

--2 x 10^6 gpd pine caustic extraction filtrate processed;
--corrugated-spacer membrane cartridges used;
--precoat filtration needed; and
--membrane flux of 18 gal./day-ft^2.

(ii) Ultrafiltration System Costs. For this case costs increase due to (1) increased membrane costs, and (2) increased feed circulation rate through the membrane spirals. The costs for the neutralization and filtration section, other equipment and building, and design and administrative costs remain unchanged. Only the ultrafiltration section costs must be changed.

As before, 510 membrane modules are required, but each is more expensive. The net feed flow to the membrane plant is 1,520 gpm. Since each module requires 15 gpm, the first stage can be operated only once-through if it contains 102 modules. Instead of limiting the number of modules in Stage 1 to this number, recirculation will be employed. This requires an additional circulation pump for Stage 1.

Ultrafiltration design factors and modified costs are given in Table 20. The appropriate flow schematic is given in Figure 57.

Capital Cost Summary, Case 2.

	Cost
Filtration and neutralization section (from Case 1)	$100,000
Ultrafiltration section (includes membranes @$275,400)	$529,000
Other equipment and building (from Case 1)	$288,000
Design, Administration, and Supervision (from Case 1)	$300,000
	$1,217,100
Contingency, @ 10%	121,700
TOTAL	$1,338,800

Total utilized energy is 509 hp, excluding the flushing pump which is run only when a circulation pump is stopped.

c. Case 3 Design

Design Bases. The design bases for Case 3 are given below:

- --2 x 10^6 gpd of pine caustic extraction filtrate processed;
- --corrugated-spacer membrane cartridges used;
- --no precoat filtration required; and
- --membrane flux of 18 gal./day-ft^2.

This case is similar to Case 2, but since corrugated-spacer membrane cartridges are used, it has been assumed that precoat filtration is not required. Instead, a simple screen, such as a Bauer Hydrosieve, will be employed in place of the precoat and scavenger filters. Feed neutralization and mixing is still required. Cost savings are achieved from (1) elimination of expensive filtration equipment; and (2) the building cost is reduced by 15% ($7200). Thus, the capital costs for Case 3 will be $80,000 less than Case 2. The total

TABLE 20

ULTRAFILTRATION SECTION DESIGN DETAILS AND COSTS--CASE 2

Stage Number	1	2	3	4
Number of Modules	200	120	120	70
Circulation Rates (gpm)	3000	1800	1800	1050
Pump Differential (psi)	40	40	40	40
Utilized Horse Power	100	70	70	40
Efficiency for Estimating h.p.	70%	60%	60%	61%
Approximate Pump Size	8x10-13	6x8-13	6x8-13	6x8-11
Pump Cost (Driven, Started and Installed, but without Electrical Connections)	$4000	$3,500	$3,500	$2,800

TABLE 20
(continued)

ULTRAFILTRATION SECTION DESIGN DETAILS AND COSTS--CASE 2

Additional Equipment

Flush Pump:	1800 gpm, 40 psi, 60 HP. (note that Stage 1 must be flushed in two halves to obtain adequate flow)	$ 3,500.
Permeate Pump:	1600 gpm, 40 psi, 60 HP.	$ 3,200.
Feed Pump:	1520 gpm, to 90 psi, 125 HP. (64% efficiency)	$ 4,700.
Water Store:	20,000 gallons, installed	$10,000.
Flush and drain piping installed		$ 9,000.

SUMMARY OF ULTRAFILTRATION SECTION COSTS

Membranes, (510 x $930) (includes $275,400 for membrane cartridges)	$474,300
Pumps	25,200
Tanks and Piping	19,000
Electrical connections for a total of 485 HP to 7 pumps (transformer excluded)	10,500
TOTAL	$529,000

capital cost is then $1,258,000. This cost includes membranes at $275,400. The total energy used is 447 hp.

d. Case 4 Design

Design Bases. The design bases for Case 4 are the same as those presented for Case 1 with the exception that a depth filter system is used in place of the precoat filter system. No changes in capital requirements have been included. The capital estimates from this case are used to display the operating cost changes. The capital cost for Case 4 is $1,228,900. Total utilized energy is 374 hp.

e. Case 5 Design

Design Bases. Design bases for Case 5 are:

- --1 x 10^6 gpd pine caustic extraction filtrate processed;
- --mesh-spacer membrane cartridges used;
- --depth filtration system used; and
- --membrane flux of 18 gal./day-ft^2.

This case is similar to Case 1, but the system is designed to handle 1 x 10^6 gpd rather than Case 1 design of 2 x 10^6 gpd. In addition, the system design uses a depth filter in place of the precoat filter system used in Case 1. The capital cost estimate for Case 5 has been developed from the Case 1 capital estimate by scaling as indicated below:

CAPITAL COST SUMMARY

	Case 1 Capital Cost	Scale Factor	Case 5 Capital Cost
Filtration and neutralization section	$ 100,000	$\frac{1}{\sqrt{2}}$	$ 70,800
Ultrafiltration section	429,000	$\frac{1}{1.9}$	225,800
Other Equipment	240,000	$\frac{1}{2}$	120,000

CAPITAL COST SUMMARY (continued)

	Case 1 Capital Cost	Scale Factor	Case 5 Capital Cost
Building	48,000	$\frac{1}{1.6}$	30,000
Design, Administration and Supervision	300,000		250,000
Total			$700,600
Contingency, @ 10%			70,000
TOTAL			$770,600

Total utilized energy is 187 hp, exluding the flushing pump which is run only when a circulation pump is stopped.

f. Summary of Capital Cost Estimates

A summary of the capital cost projections for the five case studies for battery-limit plants to treat pine caustic extraction filtrate is presented in Table 21. Because a plant for this service would be the first of its kind, conservative estimates have been used in preparation of the projections. This is especially so in the estimates for design, administration and supervision. It is felt that the startup and first year supervision should be amply provided for. As will be noted, in Case 5 this accounts for about one-third of the capital costs.

These capital values are used in the following sections in the development of the projected plant operating costs.

3. Operating Cost Summary

Projected operating costs for Cases 1 through 5 are presented in Tables 22, 23, 24, 25, and 26 and are summarized in Table 27.

Bases for Estimates. The estimates have been developed on the basis of a 365-day operating year. The costs are incremental operating costs for treating pine caustic extraction filtrate in an existing pulp mill complex.

TABLE 21

SUMMARY OF INSTALLED CAPITAL COST ESTIMATES
PLANT TO TREAT PINE CAUSTIC EXTRACTION FILTRATE

Case No.	1	2	3	4	5
Filtration & Neutralization Section	$ 100,000	$ 100,000	$ 35,000	$ 100,000	$ 70,800
U.F. Membrane Section	429,000	529,000	529,000	429,000	225,800
Other Equipment	240,000	240,000	240,000	240,000	120,000
Building	48,000	48,000	40,000	48,000	30,000
Design, Administration & Supervision	300,000	300,000	300,000	300,000	250,000
Subtotal	1,117,200	1,217,100	1,114,000	1,117,200	700,600
Contingency	111,700	121,700	114,000	111,700	70,000
Total	$1,228,900	$1,338,800	$1,258,800	$1,228,900	$770,600
Membrane Costs Included in Capital	$ 183,600	$ 275,400	$ 275,400	$ 183,600	$ 91,800
Utilized HP	374	509	447	374	187

TABLE 22

Operating Costs for Treatment of Pine Caustic Extraction Filtrate, Case 1

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid	8,000 lb/day	1¢/lb	$ 80.00			
Filter Aid	1,000 lb/day	3¢/lb	30.00			
Total Material.			$110.00	$ 40,150	5.5	14.3
Conversion Expense						
Labor (including benefits)	5 Man-yrs	$11,800/yr	$161.64	$ 59,000	8.08	21.0
Repair and Maintenance						
Material	$ 744,300	1.5%/yr	30.58	11,164	1.52	3.9
Labor	0.5 Man-yrs	$20,000/yr	27.40	10,000	1.37	3.6
Electric Power	374 HP	0.746¢/HP-hr	66.96	24,400	3.35	8.7
Insurance and Taxes	1/2 x Maintenance Material		15.29	5,580	0.76	2.0
Total, excluding Depreciation			$301.87	$110,183	15.08	39.2
Depreciation						
Membranes	$ 183,600	3-year life	$167.67	61,200	8.38	21.8
Other Facilities	$1,044,300	15-year life	190.74	69,620	9.54	24.8
Total Conversion Expense			$660.28	$241,002	33.00	85.7
Total Incremental Cost			$770.28	$281,152	38.50	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			2	730		

TABLE 23

Operating Costs for Treatment of Pine Caustic Extraction Filtrate; Case 2

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid.	8,000 lb/day	1¢/lb	$ 80.00			
Filter Aid.	1,000 lb/day	3¢/lb	30.00			
Total Material			$110.00	$ 40,150	5.50	12.5
Conversion Expense						
Labor (including benefits)	5 Man-yrs	$11,800/yr	$161.64	$ 59,000	8.08	8.3
Repair and Maintenance						
Material.	$ 763,400	1.5%/yr	31.37	11,450	1.57	3.6
Labor	0.5 Man-yrs	$20,000/yr	27.40	10,000	1.37	3.1
Electric Power	509 HP	0.746¢/HP-hr	91.13	33,203	4.56	10.3
Insurance and Taxes. .	1/2 x Maintenance Material		15.69	5,725	0.78	1.8
Total, excluding Depreciation			$327.23	$119,440	16.36	37.1
Depreciation						
Membranes	$ 275,400	3-year life	$251.50	91,800	12.57	28.5
Other Facilities	$1,063.400	15-year life	194.23	70,893	9.71	22.0
Total Conversion Expense			$772.96	$282,130	44.14	100.0
Total Incremental Cost			$882.96	$322,280	44.14	
Statistical						
Effluent Treated (x 10^6 gallons)			2	730		

TABLE 24

Operating Costs for Treatment of Pine Caustic Extraction Filtrate; Case 3

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid.	8,000 lb/day	1¢/lb	$ 80.00			
Filter Aid						
Total Material			$ 80.00	$ 29,200	4.0	10.1
Conversion Expense						
Labor (including benefits)	4 Man-yrs	$11,800/yr	$129.32	$ 47,200	6.47	16.4
Repair and Maintenance						
Material	$ 682,400	1.5%/yr	28.04	10,236	1.40	3.4
Labor	0.5 Man-yrs	$20,000/yr	27.40	10,000	1.37	3.5
Electric Power	447 HP	0.746¢/HP-hr	80.03	29,211	4.00	10.1
Insurance and Taxes	1/2 x Maintenance Material		14.02	5,118	0.70	1.8
Total, excluding Depreciation			$278.81	$101,765	13.94	35.3
Depreciation						
Membranes	$ 275,400	3-year life	$251.50	91,800	12.57	31.8
Other Facilities	$ 982,400	15-year life	179.43	65,493	8.97	22.7
Total Conversion Expense			$709.74	$259,055	35.48	89.9
Total Incremental Cost			$789.74	$288,255	39.48	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			2	730		

TABLE 25

Operating Costs for Treatment of Pine Caustic Extraction Filtrate: Case 4

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid	8,000 lb/day	1¢/lb	$ 80.00			
Filter Aid						
Total Material			$ 80.00	$ 29,200	4.0	11.3
Conversion Expense						
Labor (including benefits)	4 Man-yrs	$11,800/yr	$129.32	$ 47,200	6.47	18.3
Repair and Maintenance						
Material	$ 744,300	1.5%/yr	30.58	11,165	1.52	4.3
Labor	0.5 Man-yrs	$20,000/yr	27.40	10,000	1.37	3.9
Electric Power	374 HP	0.746¢/HP-hr	66.96	24,400	3.35	9.5
Insurance and Taxes	1/2 x Maintenance Material		15.29	5,580	0.76	2.1
Total, excluding Depreciation			$269.55	$ 98,386	13.47	38.1
Depreciation						
Membranes	$ 183,600	3-year life	$167.67	61,200	8.38	23.7
Other Facilities	$1,044,300	15-year life	190.74	69,620	9.54	27.0
Total Conversion Expense			$627.96	$229,205	31.39	88.7
Total Incremental Cost			$707.96	$258,405	35.39	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			2	730		

TABLE 26

Operating Costs for Treatment of Pine Caustic Extraction Filtrate: Case 5

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid	4,000 lb/day	1¢/lb	$ 40.00			
Filter Aid						
Total Material			$ 40.00	$ 14,600	4.00	8.7
Conversion Expense						
Labor (including benefits)	4 Man-yrs	$11,800/yr	$129.32	$ 47,200	12.93	28.2
Repair and Maintenance						
Material	$ 364,800	1.5%/yr	15.00	54,720	1.5	3.3
Labor	0.5 Man-yrs	$20,000/yr	27.40	10,000	2.7	6.0
Electric Power	187 HP	0.746¢/HP-hr	33.48	12,220	3.35	7.3
Insurance and Taxes	1/2 x Maintenance Material		7.50	27,360	0.75	1.6
Total, excluding Depreciation			$212.70	$ 77,636	21.27	46.5
Depreciation						
Membranes	$ 91,880	3-year life	83.84	30,600	8.38	18.3
Other Facilities	$ 664,800	15-year life	121.42	44,320	12.14	26.5
Total Conversion Expense			$417.94	$152,548	41.79	91.3
Total Incremental Cost			$457.94	$167,100	45.79	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			1	365		

TABLE 27

DAILY INCREMENTAL OPERATING COSTS
TREATMENT OF PINE CAUSTIC EXTRACTION FILTRATE

Case No.	1	2	3	4	5
Pine Caustic Extraction Filtrate Treated x 10^6 gpd	2	2	2	2	1
Capital Investment	$1,228,900	$1,338,800	$1,258,800	$1,228,900	$770,600
Daily Operating Costs - Dollars/day					
Materials	$110	$110	$ 80	$ 80	$ 40
Conversion Expense less Depreciation	301.87	327.23	278.81	269.55	212.70
Membrane Depreciation	167.67	251.50	251.50	167.67	83.84
Other Facilities Depreciation	190.74	194.23	179.43	190.74	121.42
Total	$770.28	$882.96	$789.74	$707.96	$457.94
Costs ¢/1000 gal. Treated	38.50¢	44.14¢	39.48¢	35.39¢	45.79¢
Costs $/Ton Pine Pulp	$ 0.962	$ 1.103	$ 0.987	$ 0.085	$ 0.572

These costs are presented as representing steady-state operating costs for an ultrafiltration plant using cellulose acetate membranes from the second year of operation and on. As indicated previously, in preparation of the capital costs, since this plant would be the first of its kind and scale, due allowance has been made for special startup and supervisory costs for the first year of operation.

The bases for estimating costs were as follows:

> Materials: Acid costs and filter aid costs are present plant or vendor estimates.
>
> Labor: Labor costs have been estimated on the basis of 1973 estimated base salaries plus 30% added to cover benefits. It is assumed the plant can be operated with 4 or 5 man-years per year of operator time for the first year or so, and that this manpower level could be substantially reduced as operating experience is gained. For these estimates, however, 4 or 5 operators are used.
>
> Electric power: Electric power costs are estimated on the basis of power costs of 1¢/kw-hr.
>
> Depreciation: The membrane life is taken as 3 years and the costs are depreciated over this time period on a straight-line basis. The remainder of the plant is depreciated on a straight-line basis over a 15-year period.

A summary of the daily operating costs in presented in Table 27.

A comparison of Cases 1 and 2 shows that an operating cost increase of approximately 6¢/1000 gals. is incurred if corrugated-spacer membrane cartridges are used instead of the mesh-spacer cartridges. Examination of Cases 1 and 3 shows that there is only about a 1¢/1000 gals. cost increase if the corrugated-spacer membrane cartridges can be used without extensive prefiltration. The major difference in operating cost between Cases 2 and 3 is the savings in labor for Case 3 since only four man-years of operating labor are provided.

Case 4 considers the use of mesh-spacer cartridges but installing backwashing filters with automatic cleaning cycles. For this case, filter aid would not be required and four men would operate the system. The capital cost of these filters has been assumed to be equal to the capital cost of precoat filters, but operating costs are reduced by elimination of filter aid and a labor component. Some pilot plant experience has been obtained with such a filter--the Hydromation granuar PVC backwashing filter. The model tested filtered adquately but was not used extensively in the pilot plant since its capacity was too small. Case 4 shows operating costs for a system with a Hydromation-type filter and with mesh-spacer cartridges. It is seen that a savings of approximately 3¢/1000 gals. over Case 1 can be obtained.

Both the capital and operating costs for an ultrafiltration system are strongly dependent on the volume processed. It has been observed in the pilot plant program that the concentration of color bodies and organics in the pine caustic extraction filtrate varies substantially from day-to-day, and even hour-to-hour. In principle, it should be possible to control the pine caustic extraction filtrate flow rate such that the concentration of contaminants is at the maximum allowable level in terms of obtaining bleached pulp of acceptable quality. Through this means it is thought that the flow of pine caustic extraction filtrate can be substantially reduced from 2×10^6 gpd, possibly to as low a volume as 1×10^6 gpd. By this means, the cost of waste treatment per unit of pulp produced can be substantially reduced, even though the treatment cost per unit of effluent may increase. In Case 5, it has been assumed that the total flow of pine caustic extraction filtrate can be reduced to 1×10^6 gpd. Mesh spacers are used, and a backwashing filter is employed. Capital costs for this plant have been scaled from the 2×10^6 gpd plant using the scaling factors presented on page 148. Case 5 is to be compared to Case 4. Although the cost per unit effluent is higher in Case 5, the cost per unit pulp produced is substantially lower.

4. Some Economic Evaluations

The capital cost estimates indicate that an ultrafiltration plant to treat 2×10^6 gpd of pine caustic extraction filtrate would have an installed cost of \$1.2-\$1.4 million based on present equipment and labor costs. A similar plant to treat 1 x 10^6 gpd would cost almost 750 to 800 thousand dollars.

The operating cost estimates indicate that the most significant cost items are membrane depreciation, other facilities depreciation, operating labor and materials--which account for 70% to 90% of the daily costs in the cases presented.

The most sensitive single cost factor displayed is the total flow of caustic extraction filtrate to be treated. If the flow of this material can be limited by judicious bleachery process flow control to 1 x 10^6 gpd, the capital cost of the membranes would be reduced to one-half, the total plant cost would be almost 60% and the daily operating costs would decrease from a level of \$700 - \$800 per day to about \$450 per day.

Membrane flux is a second important parameter in process costs. The cases examined above have not considered variation in membrane flux, but have assumed a rate of 18 gal./day-ft^2 . For a given capacity, plant cost will vary almost inversely with membrane flux. Operating costs will be greatly affected due to both membrane replacement cost and capital depreciation. The value chosen in the design calculations (18 gal./day-ft^2) is higher than that observed in most of the pilot plant tests. However, since some of the membrane cartridges exhibited this ultrafiltration rate, it is assumed to be an achievable value. In addition, future advances in membrane technology will undoubtedly provide higher-flux membranes.

Membrane life is very important since membrane replacement is a substantial operating cost factor. The 3-yr life which has been chosen for design calculations is longer than any life demonstrated to date with pine caustic extraction filtrate. This life, however, is not unreasonable for brackish water desalination applications, and is considered realistic. It may be noted that a 2-yr membrane life (mesh-spacer cartridges) would increase operating costs by approximately 4¢/1000 gals. Again, with new developments in membrane technology, longer life should be obtainable.

Other anticipated membrane improvements would permit operation at alkaline pH's and at high temperatures. Three such membranes are in an advanced stage of development. These are the NS-1 membrane, developed by the North Star Research and Development Institute, Minneapolis, Minnesota (29),the polybenzimidazole membrane, developed by the Celanese Research Company, Summit, New Jersey(30),and the dynamic membranes developed by the Oak Ridge Natural Laboratory (16).Not only can acid costs and cooling costs be eliminated, but membrane flux should also increase. This is due to two factors. First, membrane flux increases with temperature; second, in the treatment of pulp mill effluents membrane flux generally decreases when the feed pH is changed. This is primarily a fouling phenomenon, with non-neutralized feeds exhibiting a substantially reduced fouling rate. Thus, new membranes will not only reduce pre-treatment costs, but can also reduce capital and operating cost since a higher membrane flux can be realized.

When using cellulose acetate membranes, for which pretreatment is required, some additional cost savings may be realized. First, the acid cost for neutralization may be reduced or eliminated by neutralizing with waste acid available within the mill. A second materials cost which may be eliminated is that for filter aid. If corrugated-spacer cartridges can be used without precoat filtration, or if a backwashable depth filter is adequate, filter aid would not be required.

Another major operating cost is labor. It has been assumed that four or five man-years/year would be required to run the combined pre-treatment and ultrafiltration systems. If the system is simplified by modification or elimination of the pre-treatment system, this operating labor load could be reduced substantially. Furthermore, as operating experience is gained, the labor requirement should decrease.

Thus, several potential savings in both capital and operating costs can be obtained through new developments in membrane technology, as well as by obtaining operating experience for a full-scale plant.

B. DECKER EFFLUENTS

1. Flow Schematic

Figure 4 (page 21) has presented a generalized flow schematic showing integration of an ultrafiltration system in the decker and black liquor systems. A generalized flow schematic, more specific to the ultrafiltration process, is shown in Figure 58. The effluent from the decker undergoes pre-treatment (neutralization and filtration), is cooled by exchange with incoming fresh water, and flows into the ultrafiltration system. The permeate from the ultrafiltration system is recycled to the final stage of pulp washing, or is used for other pulp mill water requirements. The concentrate from the ultrafiltration section is used to sluice the filter cake, and this suspension is processed in the black liquor recovery system.

More detailed flow schematics for the decker effluent treatment system are given in Figures 56 and 57 (pages 129 and 130).These drawings, previously presented for the pine caustic extraction filtrate system, are applicable to the treatment system for decker effluents. Only the means of disposal of the permeate and concentrate fractions from the ultrafiltration system are different.

In preparation of the five cases of projected economics presented, the process parameters have been varied as shown in Table 28.

2. Capital Cost Projections

The five capital cost projections for installed battery-limit plants are presented in Table 29. The vendor equipment budgetary estimates used in preparation of the pine caustic extraction filtrate capital projections have been used as the basis for the estimates and are not repeated here.

Cases 6 and 9 differ from the capital estimate for Case 1 (pine caustic extraction filtrate) only by the cost of the evaporator system used in Case 1. Details for the costs are given under the Case 1 discussion.

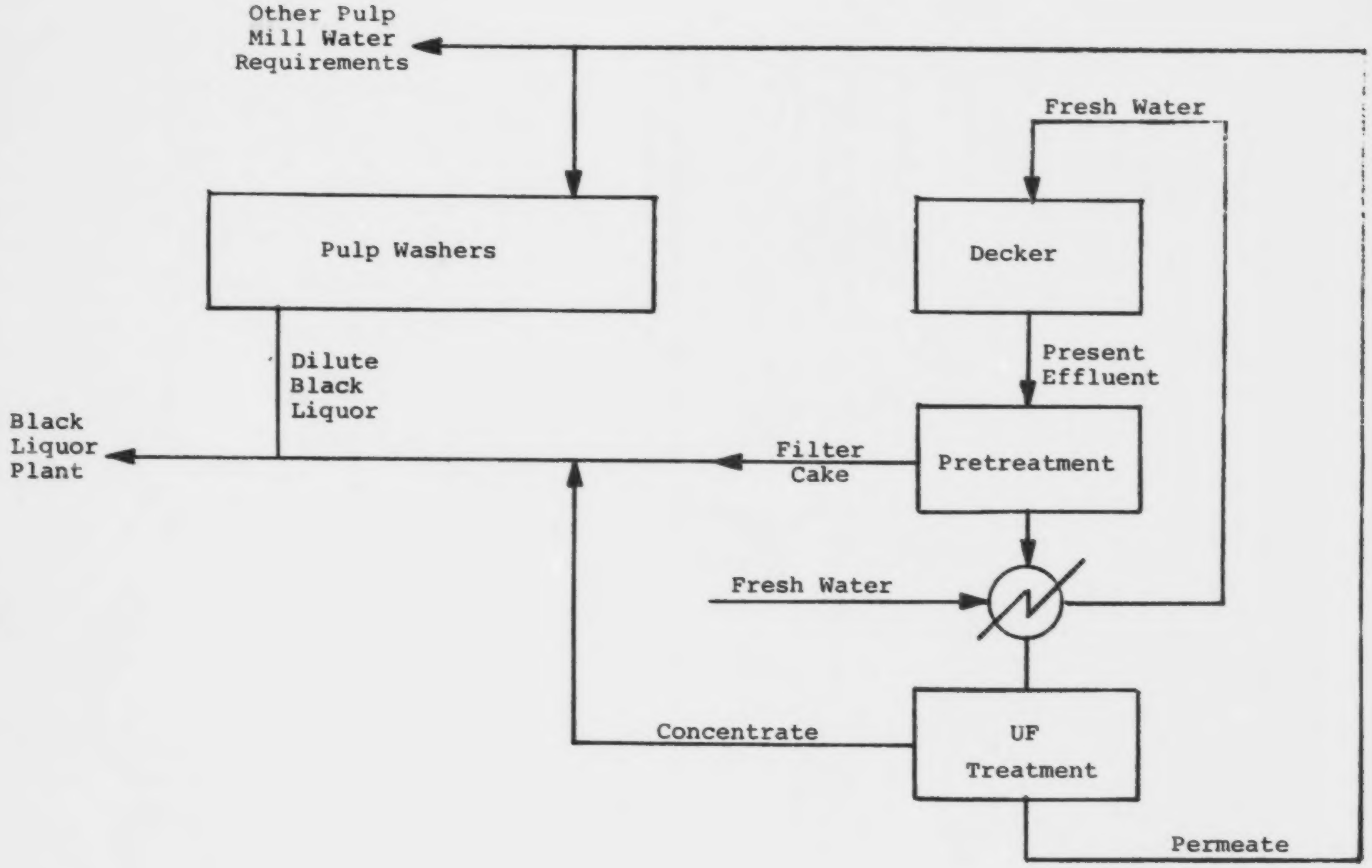

Other Pulp
Mill Water
Requirements
Fresh Water
Pulp Washers
Decker
Dilute
Black
Liquor
Present
Effluent
Black
Liquor
Plant
Filter
Cake
Pretreatment
Fresh Water
Concentrate
UF
Treatment
Permeate

FIGURE 58: SIMPLIFIED FLOW SCHEMATIC: TREATMENT OF DECKER EFFLUENTS

TABLE 28

CASES FOR CAPITAL COST ESTIMATES

DECKER EFFLUENTS

Case	Decker Effluent Flow Rate (x 10^6 gpd)	Membrane Modules: Low Flow (mesh spacer)	Membrane Modules: High Flow (corrugated spacer)	Membrane Flux gal./day-ft^2	Circulation Rate gpm/membrane cartridge	Membrane Cost $/sq ft	Prefiltration: Precoat Filter	Prefiltration: Backwashable Depth Filter	Prefiltration: Screen Filter	Operating Manpower No. of Men
6	2	x		18	8	$1.50	x			5
7	2		x	18	15	$2.25			x	4
8	1	x		18	8	$1.50	x			5
9	2	x		18	8	$1.50		x		4
10	1	x		18	8	$1.50		x		4

TABLE 29

INSTALLED CAPITAL ESTIMATES - PLANT TO TREAT DECKER EFFLUENTS

Case No.	6[a]	7[b]	8[c]	9[a]	10[c]
Prefiltration & Neutralization Section	$100,000	$100,000 -65,000 =35,000	$\frac{\$100,000}{\sqrt{2}}$ =70,800	$100,000	$ 70,800
Ultrafiltration Membrane Section	429,100	529,000	$\frac{429,100}{1.9}$ =225,800	429,100	225,800
Other Equipment	115,000	115,000	115,000/2 =53,000	115,000	53,000
Building	48,000	40,800	$\frac{48,000}{1.6}$ =30,000	48,000	30,000
Design, Administration & Supervision	300,000	300,000	250,000	300,000	250,000
Subtotal	992,200	1,019,800	629,600	992,200	629,600
Contingency	98,200	101,000	60,000	98,200	60,000
Total	$1,090,400	$1,120,800	$689,600	$1,090,400	$689,600
Membrane Costs Included in Capital	183,600	275,400	183,600 =91,800	183,600	91,800
Utilized HP	374	447	187	374	187

NOTES:

a - costs taken from Case 1 and adjusted
b - costs taken from Case 3 and adjusted
c - costs obtained by adjustment and scaling from Case 6

Case 7 differs from Case 3 (pine caustic extraction filtrate) only by the cost of the evaporator system. Details are given under Case 3.

Case 8 and Case 10 are derived from Case 6 by the scaling procedures used under the pine caustic extraction filtrate Case 5.

3. Estimated Operating Costs

Projected operating costs for each of the 5 cases are presented in Tables 30, 31, 32, 33 and 34 . The estimates are based on a 365-day operating year.

The projected operating costs as presented are incremental operating costs for a plant for treating the decker effluents and their reuse. These costs are presented for steady state operations from the second year of operation and on. In preparation of the capital costs, since this plant would be the first of its kind and scale, due allowance has been made for special startup and supervisory costs for the first year operations.

The same estimating bases have been used for the decker effluent cases as were used in the pine caustic extraction filtrate cases. The life of the membranes is taken as 3 years and the life of the other facilities as 15 years for purposes of depreciation estimates. Labor costs as presented include a 30% adder for benefits.

The daily incremental operating costs for the five cases are summarized in Table 35. These cases are evaluated in greater detail below, which makes allowance for process credits.

4. Potential Process Credits from Decker Effluent Recycle

The design of the ultrafiltration plant to treat decker effluents is developed on the basis of splitting the stream into a concentrate containing organic materials, including color bodies, and a permeate stream of low color containing the bulk of the dissolved materials, primarily sodium sulfate. The low-volume concentrate would contain 10-20% organic material, and would be

TABLE 30

Operating Costs for Treatment of Decker Effluent; Case 6

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid.	8,000 lb/day	1¢/lb	$ 80.00			
Filter Aid.	1,000 lb/day	3¢/lb	30.00			
Total Material			$110.00	$ 40,150	5.5	14.9
Conversion Expense						
Labor (including benefits)	5 Man-yrs	$11,800/yr	$161.64	$ 59,000	8.08	21.9
Repair and Maintenance						
Material	$ 606,800	1.5%/yr	24.94	9,102	1.25	3.3
Labor.	0.5 Man-yrs	$20,000/yr	27.40	10,000	1.37	3.9
Electric Power	375 HP	0.746¢/HP-hr	66.96	24,400	3.35	9.1
Insurance and Taxes	1/2 Maintenance Material		12.26	4,476	.62	1.7
Total, excluding Depreciation.			$293.20	$107,017	14.67	39.8
Depreciation						
Membranes	$ 183,600	3-year life	$167.67	61,200	8.38	22.8
Other Facilities	$ 906,800	15-year life	165.63	60,453	8.28	22.5
Total Conversion Expense			$626.50	$228,673	31.33	85.1
Total Incremental Cost			$736.50	$268,823	36.83	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			2	730.0		

TABLE 31

Operating Costs for Treatment of Decker Effluent: Case 7

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid.	8,000 lb/day	1¢/lb	$ 80.00			
Filter Aid						
Total Material			$ 80.00	$ 29,200	4.0	10.6
Conversion Expense						
Labor (including benefits)	4 Man-yrs	$11,800/yr	$120.32	$ 47,200	6.47	17.1
Repair and Maintenance						
Material	$ 545,400	1.5%/yr	22.41	8,810	1.12	3.0
Labor.	0.5 Man-yrs	$20,000/yr	27.40	10,000	1.37	3.6
Electric Power	447 HP	0.746¢/HP-hr	80.03	29,210	4.00	10.6
Insurance and Taxes	1/2 x Maintenance Material		11.20	4,090	0.56	1.5
Total, excluding Depreciation			$270.76	98,827	1.35	35.8
Depreciation						
Membranes	$ 275,000	3-year life	$251.50	91,800	12.57	33.2
Other Facilities	$ 845,400	15-year life	154.41	56,360	7.72	20.4
Total Conversion Expense			$676.67	$246,985	$33.81	89.4
Total Incremental Cost			$756.67	$276,185	$37.81	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			2	730		

TABLE 32

Operating Costs for Treatment of Decker Effluent: Case 8

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid.	4,000 lb/day	1¢/lb	$ 40.00			
Filter Aid	500 lb/day	3¢/lb	15			
Total Material			$ 55.00	$ 20,070	5.5	11.3
Conversion Expense						
Labor (including benefits)	5 Man-yrs	$11,800/yr	$161.64	$ 59,000	16.15	33.1
Repair and Maintenance						
Material	$ 297,300	1.5%/yr	12.22	4,450	1.22	2.5
Labor.	0.5 Man-yrs	$20,000/yr	27.40	10,000	2.74	5.6
Electric Power	187 HP	0.746¢/HP-hr	33.48	12,220	3.35	6.9
Insurance and Taxes	1/2 x Maintenance Material		6.11	2,230	0.61	1.2
Total, excluding Depreciation			$240.85	$ 87,910	24.08	49.3
Depreciation						
Membranes.	$ 91,800	3-year life	$ 83.84	30,600	8.38	17.1
Other Facilities . .	$ 597,300	15-year life	109.10	39,820	10.91	22.3
Total Conversion Expense.			$433.38	$158,333	43.47	88.7
Total Incremental Cost.			$488.38	$178,259	48.87	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			1	365		

TABLE 33

Incremental Operating Costs for Treatment of Decker Effluent; Case 9

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid.	8,000 lb/day	1¢/day	$ 80.00			
Filter Aid						
Total Material			$ 80.00	$ 29,200	4.0	11.9
Conversion Expense						
Labor (including benefits)	4 Man-yrs	$11,800/yr	$129.32	$ 47,200	6.47	19.2
Repair and Maintenance						
Material	$ 606,800	1.5%/yr	24.94	9,102	1.25	3.7
Labor.	0.5 Man-yrs	$20,000/yr	27.40	10,000	1.37	4.1
Electric Power.	374 HP	0.746¢/HP-hr	66.96	24,400	3.35	9.9
Insurance and Taxes	1/2 x Maintenance Material		12.26	4,476	0.62	1.8
Total, excluding Depreciation.			$260.88	$ 95,178	13.06	38.7
Depreciation						
Membranes.	$ 183,600	3-year life	$167.67	$ 61,200	8.38	24.9
Other Facilities .	$ 906,800	15-year life	165.63	60,453	8.28	24.6
Total Conversion Expense.			$594.18	$216,831	29.72	88.1
Total Incremental Cost.			$674.18	$246,031	33.72	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			2	730		

TABLE 34

Incremental Operating Costs for Treatment of Decker Effluent; Case 10

	Quantity	Unit Cost	$/Day	$/Year	¢/M-gal	% of Total
Material						
Acid	4,000 lb/day	1¢/lb	$ 40.00			
Filter Aid						
Total Material			$ 40.00	$ 14,600	4.0	9.1
Conversion Expense						
Labor (including benefits)	4 Man-yrs	$11,800/yr	$129.32	$ 47,200	12.93	29.3
Repair and Maintenance						
Material	$ 297,300	1.5%/yr	12.22	4,460	1.22	2.8
Labor	0.5 Man-yrs	$20,000/yr	27.40	10,000	2.74	6.2
Electric Power	187 HP	0.746¢/HP-hr	33.48	12,220	3.35	7.6
Insurance and Taxes	1/2 x Maintenance Material		6.11	2,230	0.61	1.4
Total, excluding Depreciation			$208.53	$ 76,110	20.85	47.2
Depreciation						
Membranes	$ 91,800	3-year life	$ 83.84	$ 30,600	8.38	19.0
Other Facilities	$ 597,300	15-year life	$109.10	39,820	10.91	24.7
Total Conversion Expense			$401.47	$146,530	40.14	90.9
Total Incremental Cost			$441.45	$161,130	44.14	100.0
Statistical						
Effluent Treated (x 10^6 gallons)			1	365		

TABLE 35

DAILY INCREMENTAL OPERATING COSTS TREATMENT AND REUSE OF DECKER EFFLUENTS

Case No.	1	2	3	4	5
Decker Effluent Feed Rate x 10^6 gpd	2	2	1	2	1
Capital Investment	$1,090,400	$1,120,800	$689,600	$1,090,400	$689,600
Daily Incremental Operating Costs					
Materials	$110	$ 80	$ 55	$ 80	$ 40
Conversion Expense less Depreciation	293.20	270.76	240.85	260.88	208.53
Membrane Depreciation	167.67	251.50	83.84	167.67	83.84
Other Facilities Depreciation	165.63	154.40	109.10	165.63	109.10
Total	$736.50	$756.67	$488.38	$674.18	$441.45
Costs ¢/1000 gal. Treated	36.83	37.81	48.87	33.72	44.14

sent to the dilute black liquor system. The permeate would be reused, either in the pulp washers or in other fresh water pulping uses and hence would eventually be returned to the dilute black liquor stream for chemical recovery.

Operation with recycle of permeate and concentrate should result in process credits which could offset the costs associated with installation and operation of the ultrafiltration plant. Potential credits could be in the following:

- --reduction of the fresh water requirements of the pulp mill (∿3-5%);
- --reduction of the total mill effluent and waste processing operations (∿3-5%);
- --retention of the salts presently being discharged in the effluent (∿16-24 tons/day);
- --increasing the organic content of the dilute black liquor without increase in liquor volume (∿8-10 tons/day);
- --reduction of the mill effluent color by about 20%; and
- --reduction of organic solids treated in the secondary treatment plant by about 20%.

The potential credits to be derived are a function of the specific pulp mill in which such an ultrafiltration plant would be installed. The potential credits discussed below are those that might be applicable at the pulp mill in which these pilot studies were conducted. At other pulp mills the washing procedures, water costs, waste disposal costs and nature of the total mill effluent may well differ. However, it is felt that the conservative figures presented below are typical "ballpark" credits which would be available at pulp mills where the decker effluents are presently sewered.

a. Water Credits

At present, water at the Canton Mill costs about $\$110/10^6$ gal. for fresh water feed and treatment of the water in the waste treatment operation. The potential water credit, then, is a function of the actual amount of water used. In this study, two cases have been examined. At 2 x 10^6 gpd recycle flow, the credit would be 2 x 110 = $220 per day. At 1 x 10^6 gpd recycle flow the credit would be $110 per day. (It should be noted those water costs are low compared to many mills because of geographical location and well planned waste disposal facilities.)

In the mill the water recycle would reduce the fresh water requirements and also the effluent handling requirements by 3-5%. No direct credit is taken for this physical reduction of volume flow in this presentation.

b. Retention of Salts

Recycling both the concentrate and the permeate fractions of the decker effluents will retain all the contained materials presently going to the waste treatment plant. Depending on the operating conditions, the decker effluents contain 1200-3300 ppm of sodium sulfate. For the calculation below, a value of 2500 ppm at 2 x 10^6 gpd flow is taken. A value of 1¢/# is assumed for the sodium sulfate value.

Potential credit for retained salts =

$$(2500 \times 10^{-6})(2 \times 10^{6})(8.33)(0.01) = \$416$$

Recycle of these materials, of course, will reduce the dissolved salt content of the total effluent. No credit is taken for this result.

c. Organic Content

About 98% of the organic content of the decker effluent is in the concentrate stream. This stream will contain >10% organic material. The heating value of this material is assumed to be 8000 BTU/#. Because no total water increase in the weak black liquor stream is anticipated as a result of the recycle and reuse of the water, the total heat value of the retained organics is taken as a potential credit.

The organic content is taken as 1000 ppm at a 2×10^6 gpd flow. The heat input is assumed to be worth 50¢/10^6 BTU. Then,

Potential heat value credit =

$$(1000 \times 10^{-6})(2 \times 10^{6})(8.33)(8000)(0.50 \times 10^{-6}) =$$

$66.6

The decker effluents exit temperatures are 120-125°F. Returning this stream to the process has two effects. The thermal balance of the pulping process is improved and the thermal load on the mill effluent is decreased. No credit is taken for either effect in this calculation.

d. Total Potential Credits

The potential credits from installation of the process are totaled below. In the calculations it has been assumed that there is a basic load of material to be removed in the washing operations and that the effect of varying the washing volumes will be to change the concentration of these materials, but that the total amount removed will remain the same.

For 2×10^6 gpd flow the total potential process credits are $220 + 416 + 67 = $703 per day.

For 1×10^6 gpd flow the total potential process credits are $110 + 416 + 67 = $593/day.

5. An Evaluation of Process Economics

The capital costs of the ultrafiltration plants for treating the decker effluents are those for treating the pine caustic extraction filtrate, modified by elimination of the concentrate disposal systems required for the latter. Consequently the capital costs and the daily operating costs displayed in Tables 29 and 35 are lower than for similar cases in the pine caustic extraction filtrate treatment.

Again the most sensitive parameter in the study is the flow of the decker effluent stream. For example, Case 9 (2×10^6 gpd) has a capital cost of $1,090,400 and a daily operating cost of $674.18; Case 10, which presumably represents the same level of treatment but on a more concentrated stream of 1×10^6 gpd, has a projected capital cost of $689,600 and a daily operating cost of $441.45.

As in the previous study the other parameters which are highly sensitive are membrane flux and life, capital depreciation, operating manpower, and materials.

The decker effluent cases differ from the pine caustic extraction filtrate cases in that for the former the potential credits defray the operating costs of the treatment plant. On the assumption that the projected capital and operating costs and potential credits are realistic, a decker effluent treatment plant, depending on size and process configuration, would have either a small net operating cost (comparison of $736.50/day, operating cost, Case 6, and $703/day potential credit), or a small net credit (comparison of $441.45/day, operating cost, Case 10, and $593/day potential credit). Consistent with the precision of the cost estimates and knowledge of the plant flow volumes, it is likely that an ultrafiltration plant to treat decker effluents could be a low-cost or net-credit operation. The process could produce color and BOD reduction and plant flow reduction values which have not been treated as credits in this evaluation.

It must be emphasized that the attractive steady state operating economics presented here are projections from the pilot operation. As in the previous set of projections for the pine caustic extraction filtrate these economics require additional experimental engineering verification, especially in the area of:

--quantifying the minimum controllable flow of decker effluents which can be used while producing a pulp of acceptable quality for subsequent mill operations;

--demonstrating that reliable membranes can be obtained commercially;

--evaluating alternative plant designs, including additional experimental work, to assure that the plant is capable of continuous 365-day operation with minimum capital and operating costs, especially labor.

The technical feasibility of treating the decker effluents has been demonstrated using the feed treatment and ultrafiltration processes described. It would be premature, at present, however, to proceed with a full-scale plant design until the information indicated above is obtained.

SECTION VII

WATER REUSE

One potential value to be obtained from the use of an ultrafiltration process to treat pulp mill process effluents is to provide a permeate which can be reused within the mill. This would reduce the fresh water requirements, as well as the total plant effluent volume.

During the course of the program, as permeate samples were produced and analyzed, evaluations of water reuse potential were performed.

A. PINE CAUSTIC EXTRACTION FILTRATE PERMEATE

The pilot plant produced permeates which had 90-96% of the color removed and which represented 99-99.5% of the flow stream fed to the system. The permeate contained most of the non-organic dissolved materials (salts) and a residual color of usually greater than 1000 ppm--even with the high color removal effectiveness (see Appendix for representative values).

Because of the stream color and high dissolved material content (usually ∿ 6000 ppm) it is felt that this permeate would have limited usefulness in the pulping and bleaching process areas. At best, it is felt, this permeate could be used to augment the volumes of other "dirty" water presently used in operations such as wood washing and cleaning of fly ash collectors. No high value reuse capability has been developed for this permeate.

B. DECKER EFFLUENT PERMEATES

The pilot plant produced permeates from both the pine and hardwood decker effluents which had about 98-99.5% of the color removed, and which represented about 99% of the stream feed to the system. As discussed in other parts of this report, the concentrate from the system would be returned to the weak black liquor stream. The permeate with its low color (∿ 100 ppm color) and dissolved salts, primarily sodium and sulfate, is a stream of high potential value for direct multiple reuse in the washers of the pulping system, or as make up water in the pulping operations. As such, the reuse of the decker effluents at a mill such as the Canton mill would:

a) reduce the treated water demand by about 4%;
b) reduce the plant effluent by about 4%;
c) recycle 15-25 tons/day of chemicals normally lost in the decker effluents; and
d) reduce the organic loading of the biological waste treatment system by about 20%.

SECTION VIII

ACKNOWLEDGEMENTS

The support of the project by the Environmental Protection Agency and the help provided by Mr. George Webster, Mr. Edmond P. Lomasney, the Federal Grant Project Officer, Dr. Karl Suva and Ralph Scott is acknowledged with sincere thanks.

The project was directed by Dr. Henry A. Fremont of Champion International. The experimental program was supervised by Mr. Dan C. Tate of Champion International.

The membrane equipment was provided by Abcor, Inc. Drs. Robert L. Goldsmith, James R. Ryan, and David J. Goldstein, and Messrs. Sohrab Hossain and A.C.F. Ammerlaan, all of Abcor, Inc., contributed to the technical support of the project and report preparation.

The progress of the project would not have been possible without the cooperation and counsel of Champion International personnel, including John O. Parrott, Plant manager, Champion International, Canton Mill, and his very competent staff; Richard Wigger; Austin Moore; Robert Townsend; C. E. Fredericks; and, Dr. A. E. Vassiliades.

SECTION IX

REFERENCES

1. Interstate Paper Corporation for the Environmental Protection Agency, Program #12040 ENC, Grant #WPRD 183-01-68, "Color Removal from Kraft Pulping Effluent by Lime Addition," December 1, 1971.

2. Spruill, Edgar L., "Paper Mill Waste: Treatment for Color Removal," IW, March/April 1971, pp. 15, 21-23.

3. Gould, Matthew, "Lime-Based Process Helps Decolor Kraft Wastewater," Chem. Eng., January 25, 1971, pp. 55-57.

4. Tejera, N.E. and Davis, M.W., Jr., "Removal of Color and Organic Matter from Kraft Mill Caustic Extraction Waste by Coagulation," TAPPI, 53, No. 10, October 1970, pp. 1931-1934.

5. National Council for Air and Stream Improvement, Inc., "The Mechanisms of Color Removal in the Treatment of Pulping and Bleaching Effluents with Lime. I. Treatment of Caustic Extraction Stage Bleaching Effluent," Technical Bulletin No. 239, July 1970.

6. National Council for Air and Stream Improvement, Inc., "The Mechanisms of Color Removal in the Treatment of Pulping and Bleaching Effluents with Lime. II. Treatment of Chlorination Stage Bleaching Effluents," Technical Bulletin No. 242, December 1970.

7. Davis, C.L., Jr., "Tertiary Treatment of Kraft Mill Effluent Including Chemical Coagulation for Color Removal," TAPPI, 52, No. 11, November 1969, pp. 2132-2134.

8. Middlebrooks, E. J., Phillips, W.E., Jr. and Coogan, F.J., "Chemical Coagulation of Kraft Mill Wastewater," IW Water and Sewage Works Supplement, March 1969, pp. 7-9.

9. Smith, S.E. and Christman, R.F., "Coagulation of Pulping Wastes for the Removal of Color," Journal WPCF, 41, No. 2, Part 1, February 1969, pp. 222-231.

Preceding page blank

REFERENCES
(continued)

10. "Projects of the Industrial Pollution Control Branch, July 1971," Water Pollution Control Research Series # 12000-07/71, pp. 5-22, 5-23, 5-24, 5-25.

11. *Ibid*., pp. 5-13, 5-26.

12. Proceedings of the TAPPI 8th Water and Air Conference, Boston, Mass., 1971, paper entitled "Activated Carbon System for Treatment of Paper Mill Washwaters."

13. *Ibid*., "Color Removal from Kraft Bleach Wastes by Ion Exchangers."

14. "Carbon Treatment of Kraft Condensate Wastes," *TAPPI*, *51*, 241, 1968.

15. Lacey, R.E. and Loeb, S., eds., *Industrial Processing with Membranes*, Wiley-Interscience, New York, 1972, pp. 223+.

16. Moore, G.E. Minturn, R.E., et. al., "Hyperfiltration and Cross-Flow Filtration of Kraft Pulp Mill and Bleach Plant Wastes," ORNL-NSF-EP-14, May, 1972.

17. Wiley, A.J., Dubey, G.A. and Bansal, I.K., "Reverse Osmosis Concentration of Dilute Pulp & Paper Effluents," for the Environmental Protection Agency, Project #12040 EEL, February, 1972.

18. Morris, D.C., Nelson, W.R. and Walraven, G.O., "Recycle of Papermill Waste Waters and Application of Reverse Osmosis," for the Environmental Protection Agency, Program #12040, January, 1972.

19. Bansal, I.K., Dubey, G.A. and Wiley, A.J., "Development of Design Factors for Reverse Osmosis Concentration of Pulping Process Effluents," presented at Membrane Symposium, National Meeting of American Chemical Society, Chicago, Illinois, September 14-18, 1970.

20. Beder, H. and Gillespie, W.J., "Removal of Solutes from Mill Effluents by Reverse Osmosis," *TAPPI*, *53*, No. 5, May 1970 , pp. 883-887.

REFERENCES
(continued)

21. Bregman, Jacob I., "Membrane Processes Gain Favor for Water Reuse," Environmental Science & Technology, 4, No. 4, April 1970, pp. 296-302.

22. Wiley, A.J., Dubey, G.A., Holderby, J.M. and Ammerlaan, A.C.F., "Concentration of Dilute Pulping Wastes by Reverse Osmosis and Ultrafiltration," Journal WPCF, 42, No. 8, Part 2, August 1970, pp. R279-R289.

23. Ammerlaan, A.C.F. and Wiley, A.J., "Pulp Manufacturers Research League Demonstrates Reverse Osmosis Process," TAPPI, 52, 1969.

24. Ammerlaan, A.C.F. and Wiley, A.J., "The Engineering Evaluation of Reverse Osmosis as a Method of Processing Spent Liquors of the Pulp and Paper Industry," prepared for the New Orleans Meeting of A.I.Ch.E., March 17-20, 1969.

25. Ammerlaan, A.C.F., Lueck, B.F. and Wiley, A.J., "Membrane Processing of Dilute Pulping Wastes by Reverse Osmosis," TAPPI, 52, No. 1, January 1969, pp. 118-122.

26. "Industrial Ultrafiltration," in Membrane Processes in Industry and Biomedicine, Plenum Press, 1971.

27. Flinn, J.E., ed., Membrane Science and Technology, Plenum Press, New York, 1970, pp. 33+, 47+.

28. Foreman, G.E., et al., "The Improvement of Spiral-Wound Reverse Osmosis Membrane Modules," Research and Development Progress Report, No. 675, Office of Saline Water, April, 1971, pp.13.

29. Cadotte, J.E., and Rozelle, L.T. of North Star Research and Development Institute, "In Situ-Formed Condensation Polymers for Reverse Osmosis Membranes" for Office of Saline Water, U.S. Department of the Interior, Third Quarterly Report, January 9, 1972, through April 8, 1972, Contract No. 14-30-2883.

REFERENCES
(continued)

30. Hossain, S., Goldsmith, R.L., Tan, M., Wydeven, T., and Levan, M.I., "Evaluation of 165°F Reverse Osmosis Modules for Washwater Purification", for Intersociety Conference on Environmental Systems, July 1973, San Diego.

APPENDIX A

DETAILED PILOT PLANT PROCESS DESCRIPTION

(as initially installed)

APPENDIX A

DETAILED PILOT PLANT PROCESS DESCRIPTION

(as initially installed)

A generalized description of the process was given in Section IV.A. The following information describes the pilot plant, as initially installed, in detail.

A detailed process flow is shown in Figure A-1. The process description which follows is based on normal operation. Components which were used at other times are described in Table A-I, a complete listing of all system components by intended function.

Referring to Figure A-1, the numbers contained in the diamond symbols refer to the process streams listed at the bottom of the Figure. This table gives average, maximum, and minimum flow rates at different points in the system.

Feed flow from the plant was to the 500 gal. fiberglas feed tank (T-1). Flow was through a float valve (V-7), which kept T-1 filled so long as feed was available from the mill. If feed flow were interrupted, a low level shutdown switch in the tank (LS-F) shut down the pilot plant and sounded an alarm. The unit was fitted with a Lightnin mixer (M-1) which kept the tank contents well mixed, enabling pH and temperature to be controlled accurately.

Temperature was measured and controlled by a probe installed in the tank and control unit (TIS-1), which controlled flow of cold water or steam through a heat transfer coil installed in the tank (HE-1). Although feed left the mill hot (approximately 120-135° F) some cooling occurred before the feed flow reached the feed tank. Consequently, a control system was provided to allow either heating or cooling by manual selection. An automatic shutdown switch and audible alarm was included in the temperature controller, which would shut down the system should a maximum preset temperature be exceeded. A separate temperature probe in T-1 was connected to a recorder giving a continuous record of feed temperature (TR-1).

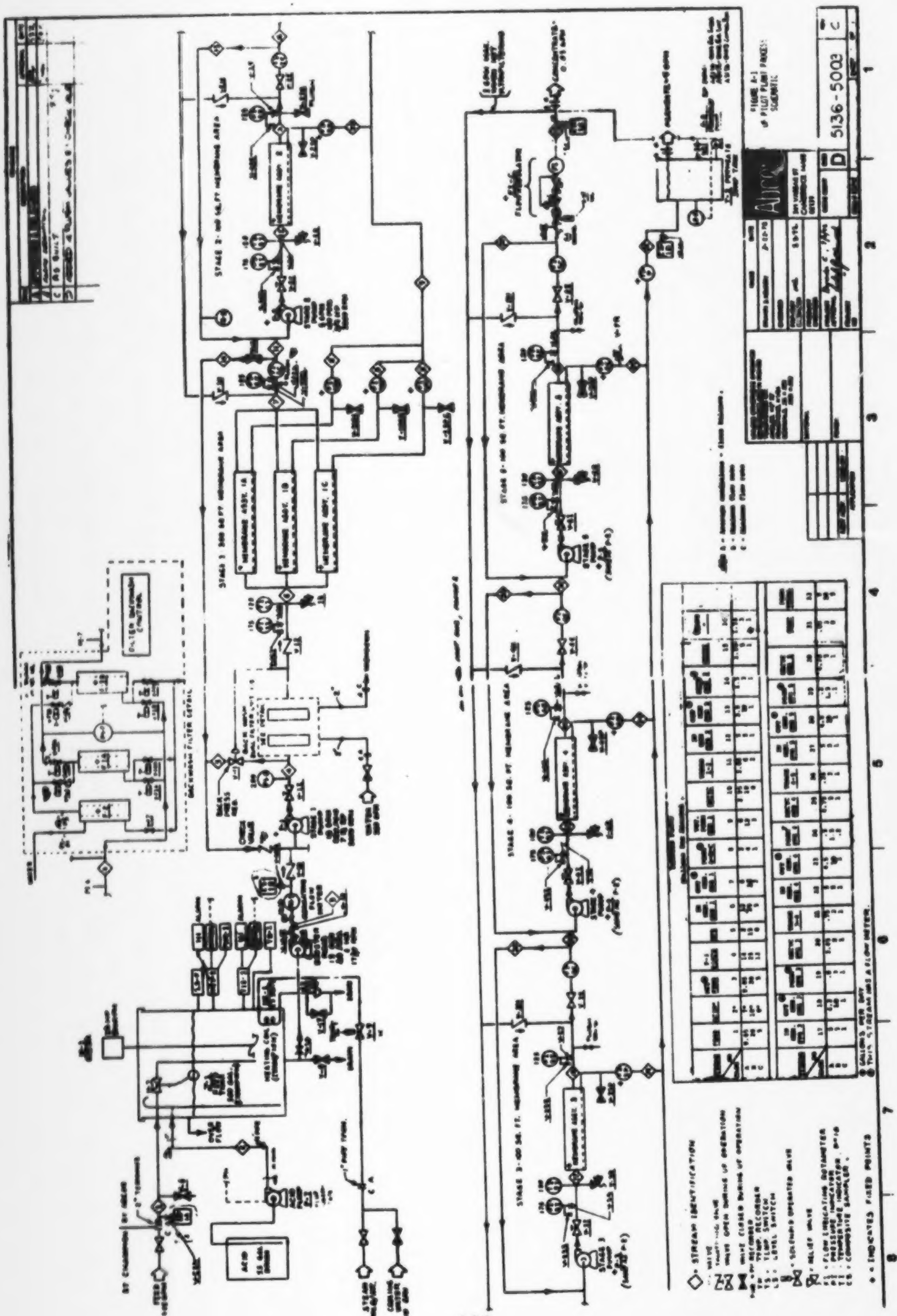


FIGURE A-1
PILOT PLANT PROCESS SCHEMATIC
Abcor
D
5136-5002
C
STAGE 1 - 300 SQ. FT. MEMBRANE AREA
STAGE 2 - 100 SQ. FT. MEMBRANE AREA
STAGE 3 - 100 SQ. FT. MEMBRANE AREA
STAGE 4 - 100 SQ. FT. MEMBRANE AREA
STREAM IDENTIFICATION
1 2 3 4 5 6 7 8

TABLE A-I

COMPONENT LIST

Code	Name	Identification	Location (assumes operator is facing control panel)	Function
CS-1	Composite Sampler	Sigmamotor Pump, Model AL-4, 4-channel finger pump	Enclosed in dog house on top of module tray	Continuously pumps small quantitites of four process streams to composite sample collectors. The four boxes labelled CS-1A, CS-1B, CS-1C, and CS-1D in Figure 2 represent channels for raw feed, neutralized feed, final concentrate, and mixed permeate, respectively.
F-1A F-1B	Process Filters	Two Broughton Corp. Model 3000 filters with 10 μ stainless steel baskets	On module, left hand side	Filters neutralize the feed prior to introduction into the membrane assemblies.
F-2	Water Filter	Broughton Corp. Model 990 filter with 200 mesh stainless steel basket	On module, left hand side	Filters water used for backwash of F-1A and F-1B.
FM-1	Integral Feed Flow Meter	Water meter	Mounted on top of barrel of P-1; on module	Records the total amount of process fluid handled by the membrane system.
FI-1C	First Stage Concentrate Flow Meter	Brooks Instrument 25 gpm rotameter, Model 1305	Control panel	Measures Stage 1 concentrate flow.

TABLE A-I
Page 2

Code	Name	Identification	Location	Function
FI-2C FI-3C FI-4C FI-5C	Concentrate Flow Meters	Brooks Instrument 10 gpm rotameters, Model 1305	Control panel	Measures concentrate flows for Stages 2-5.
FI-C	Concentrate Flow Regulator and Flow Meter	Brooks Instrument Self-contained flow controller, Model 1350-8802-5-65C	Control panel	Regulates and measures concentrate flow from Stage 5.
FI-1PA FI-1PB FI-1PC FI-2P FI-3P FI-4P FI-5P	Permeate Flow Meters	Dwyer Instruments polycarbonate rotameters, Models RMC 141,142,143	Control panel	Measures permeate flow rates from each of the seven membrane assemblies.
FI-P	Mixed Permeate Flow Meter	Brooks Instrument 25 gpm rotameter, Model 1305	Control panel	Measures flow rates of mixed permeate from all membrane assemblies.
HE-1	Heat Transfer coil	25 sq ft, 1/2" diameter titanium coil	Feed tank (T-1)	Heat transfer surface for heating or cooling of process fluid.
LS-F	Feed Tank Level Switch	Gems level switch, Model LS-1900	Feed tank (T-1)	Shuts down membrane system and sounds alarm on low level of process fluid

TABLE A-I
Page 3

Code	Name	Identification	Location	Function
LS-P	Permeate Sump Level Switch	Gems level switch, Model LS-1900	In permeate sump tank (T-2)	Shuts down permeate pressurization pump (P-8) on low level in permeate sump tank.
M-1	Feed Tank Mixer	Lightnin mixer, Model ND-2A	Feed tank (T-1)	Mixes feed tank contents allowing efficient neutralization and temperature control.
P-1	Stage 1 Feed Pump	Goulds multistage centrifugal pump, Model MB-13400	On bottom of module	Pumps process fluid through the automatic backwash filter unit and Stage 1 membrane assemblies This pump also provides feed pressurization in subsequent membrane stages.
P-2 P-3 P-4 P-5	Circulation Pumps for Stages 2,3,4, and 5	Goulds multistage centrifugal pumps, Model MB5100	On top right hand side of module	Boosts pressure of feed sequentially to Stages 2, 3,4, and 5.
P-6	Feed Booster Pump	Corcoran 1 hp centrifugal pump,	Mounted on feed tank panel	Transfers feed from feed tank to suction of P-1.
	Acid Pump	Precision Control diaphragm pump, Model 11321-71	Mounted by acid drum on mezzanine	Pumps acid from 55 gal. drum to feed tank for feed neutralization.

TABLE A-I
Page 4

Code	Name	Identification	Location	Function
P-8	Permeate Pressurization Pump	Little Giant plastic head pump, Model 1P732 (Grainger)	Mounted on underside of module tray	Transfers pressurized permeate into membrane modules to keep them pressurized and wet during system shutdown.
PHIS-1	pH Indicator/ Controller	Leeds and Northrop monitor, Model 7070-02-3-107-6-04, with pH electrodes and probe	Monitor mounted in in control panel; pH probe assembly in feed tank	Measures and controls pH of the process fluid. An electrode probe assembly is mounted in the feed tank and input is transmitted to the control panel. The low alarm on the monitor is used to turn the acid pump on and off with a dead band range of 0.5 pH unit. The high alarm of the monitor is used to shutdown the system and sound an alarm on rising pH.
PHR-1	pH Recorder	Rustrak Instrument millivolt recorder	Control panel	Records pH. The millivolt output from the Leeds and Northrop controller,serves as the input to the recorder. Full-scale on the recorder (10 divisions) corresponds to the pH range 2 - 12.

TABLE A-I
Page 5

Code	Name	Identification	Location	Function
PI-F1 PI-F2	Water Filter Pressure Gauges	Broughton Pressure Gauges	On water filter	Measures pressure drop across water filter (F-2). High pressure drop during the backwash cycle indicates the need to "blow-down" the water filter.
PI-6	Process Filter Pressure Gauge	0-400 psi Pressure Gauge	On bottom of module, in the piping running from P-1 outlet to process filter inlet	Measures pressure at the filter inlet.
PI-1I PI-1E PI-2I PI-2E PI-3I PI-3E PI-4I PI-4E PI-5I PI-5E	Pressure Gauges	0-300 psi Pressure Gauges	Control panel	Measures inlet and outlet pressures to each of the five membrane stages.
PS-F	Filter Differential Pressure Switch	Static O-Ring Pressure Switch,	Mounted in module between inlet and outlet piping of process filter	Senses pressure differential across the process filter, triggering an automatic backwash cycle when the pressure drop builds up to a specified level (nominally 75 psi)
PS-2	Low Pressure Switch	Static O-Ring Pressure Switch,	Mounted in piping manifold on suction side of P2-P5; top right-hand side of module	Shuts down pumps P2, P3 P4, and P5 when their suction pressure drops below a preset value (nominally 35 psi)

TABLE A-I
Page 6

Code	Name	Identification	Location	Function
S-1	Strainer	Common Y Type Pipeline Strainer	Next to upper left-hand corner of control panel	Removes suspended solids before concentrate flow regulator to prevent plugging
S[illegible]-1	Steam Trap		On exit line of HE-1, located on Feed Tank	Removes condensed steam
T-1	Feed Tank	500 gallon fiberglas tank		Surge for process fluid, and mixing vessel for control of pH and temperature.
T-2	Permeate Sump Tank	5 gallon polyethylene tank	On top of module tray	Serves as reservoir for permeate for system pressurization during shutdown
TIS-1	Temperature Indicator/ Controller	United Electric, Model 1202 Temperature Indicating/Controller	Mounted on Feed Tank control panel	Measures and controls temperature of process fluid in feed tank. Electronics control operation of V-9 allowing either steam or cooling water to pass through HE-1 when called for by the sensing element.
TR-1	Temperature Recorder	Rustrak Instruments Temperature Recorder	Control panel	Continuously records feed temperature in Feed Tank.
TI-1 TI-2 TI-3 TI-4 TI-5	Temperature Indicators	Temperature Gauges	On module; in inlet piping to membrane stages 1,2,3,4, and 5	Indicates temperature of feed to each of the five membrane stages

TABLE A-I
Page 7

Code	Name	Identification	Location	Function
V-1I	First Stage Control Valve	1" globe valve	Lower left-hand front of module	Throttles feed flow from P-1.
V-2I V-2E V-3I V-3E V-4I V-4E V-5I V-5E	Control Valves	1/2" globe valves	Control panel	To control flows and pressures through membrane stages 2,3,4, and 5; to isolate any of stages 2,3, 4 and/or 5, as desired.
V-1SI V-1SE V-2SI V-2SE V-3SI V-3SE V-4SI V-4SE V-5SI V-5SE	Feed Sample Valves	1/4" Petcocks	Control Panel	To sample inlet and outlet feed flows to each of the five membrane stages.
V-1SPA V-1SPB V-1SPC V-2SP V-3SP V-4SP V-5SP	Permeate Sample Valves	1/4" petcocks	In permeate piping on right hand side of Module	Valves for collection of permeate samples from each of the seven membrane assemblies.
V-1R V-2R V-3R V-4R V-5R	Pressure Relief Valves	Brass Pressure Relief Valves	In piping to inlet of each of membrane stages 1 through 5	Pressure relief valves preset at 175 psig to prevent over-pressurization of any of the five membrane stages should system malfunction occur

TABLE A-I
Page 8

Code	Name	Identification	Location	Function
V-PR	Permeate Pressure Relief Valve	Brass Relief Valve	In permeate line from Stage 5	Pressure relief valve preset approximately at 50 psig, to relieve pressur if overpressurization should occur in any of the permeate piping.
V-1H V-2H V-3H V-4H V-5H	Check valves	Check-all 1/2" Union Check valves	In piping on right hand side of module	To allow pressurized permeate to pass into the membrane stages upon shutdown, and to prevent process fluid from enterin the permeate sump tank whe the system is operative.
V-6	Sample Valve	1/2" globe valve	On feed inlet piping to Feed Tank	To draw off raw feed for sampling or drainage.
V-7	Float Valve	Fisher Controls float operated valve, Type 171F	Feed tank	To maintain feed tank at the desired level so long as process feed is available.
V-8	Drain Valve	2" ball valve	On tank outlet	Drain valve to drain tank contents
V-9	Steam Solenoid Valve	Automatic Switch Co. Steam Solenoid Valve, Model 8222B24	In feed tank on inlet to HE-1	To control flow of steam or cooling water to HE-1, actuated by TIS-1.

TABLE A-I
Page 9

Code	Name	Identification	Location	Function
V-10	Check Valve	1" Union Checkall Check Valve	On suction of P-1	Prevents flow reversal into the feed tank when the system is shutdown and operating under permeate pressurization.
V-11	First Stage Pressure Relief Valve	D'Este Type V Direct-acting back-pressure regulator with external pressure sensing	Behind process filters	Maintains constant pressure at filter outlet. If pressure drops at the filter outlet due to cake build-up on the filter, V-11 closes thus reducing the amount of bypass around the filter, and increasing the inlet pressure to the filter.
V-12	Check Valve	1" Checkall Union Check Valve	In piping between filter outlet and Stage 1 inlet	Prevents flow backing-up into filters during system shutdown and pressurization with permeate.
V-13	First stage bypass valve	1" globe valve	At top of control panel	To control recirculation rate around stage 1. In normal operation it is anticipated that this valve will be closed. This is to be confirmed in actual operation.
V-14	Ball Valve	Brass ball valve	On suction side of booster pump (P-6); on feed tank	Isolation valve, for service of P-6.

TABLE A-I
Page 10

Code	Name	Identification	Location	Function
V-15	Concentrate Solenoid Valve	1/4" Asco solenoid valve, Model 8262A233	On module, in concentrate line just before FI-C	This normally-closed solenoid valve is open in operation, allowing concentrate to be removed continuously from the system. When the system is shutdown the solenoid valve closes, thus preventing pressurized permeate removal from the system through the concentrate line.
V-16	Solenoid Valve	1" normally-closed Asco solenoid valve, Model 8211B27	In piping on module just before FM-1	To prevent continued drainage of feed tank contents into the membrane system during automatic shutdown. This solenoid valve will open only when the system is operational.
V-17	Bypass valve		On outlet line of HE-1 (on tank) next to ST-1	This valve is to be opened when cooling water is passed through HE-1. This permits water flow to pass to drain without having to pass through the steam trap (ST-1).
V-18	Transfer valve	1" brass globe valve	Feed Tank, on booster pump outlet	This valve allows removal of feed tank contents after neutralization and temperature control to another system, if desired. Feed material can be pumped out this line for pilot filtration tests.

TABLE A-I
Page II

Code	Name	Identification	Location	Function
V-19	Isolation Valve	1" brass ball valve	On outlet line of booster pump (P-6), on feed tank	System isolation, as needed.
V-20	Flush valve	1/2" brass globe valve	In concentrate line from stage 5; on module behind water filter (F-2)	When open, this permits rapid flushing of all five membrane stages since the flow regulator (FI-C) can be bypassed
V-21	Permeate by-pass valve	1/4" needle valve	On bottom side of module tray	To allow continuous bleed from the permeate pump (P-8) back to the permeate sump tank (T-2), to avoid overheating P-8 when pressurized permeate is not being transferred to the membrane system.
V-22 V-23 V-24 V-25 V-26 V-27 V-28 V-29 V-30 V-31	Isolation Valves	1/4" needle valves	At inlets to pressure indicators; on rear side of control panel	To isolate pressure gauges should removal be required during operation.
V-F1A V-F1B V-F2A V-F2B	Solenoid Valves	1" Asco 2-way normally-closed brass solenoid valves, Model 8211B27	On piping to process fluid filters	To isolate process filters from feed during the backwash cycle.

TABLE A-I
Page 12

Code	Name	Identification	Location	Function
V-F3A V-F3B V-F4A V-F4B	Solenoid Valves	2" Asco 2-way normally-closed brass solenoid valves, Model 8211B82	On piping to process fluid filters	Controls flow of backwash water through the filter during backwash cycle. These four solenoid valves are on the water lines to the process filters.
V-F5A V-F5B V-F6A V-F6B	Check Valves	1" and 2" brass check valves	Installed in piping to process fluid filters	Insures positive seating of solenoid valves under reverse pressurization.
V-CSA V-CSB V-CSC V-CSD	Petcocks	1/4" Petcocks	V-CSA on feed tank inlet line, V-CSB on inlet to P-1 above P-1 barrel, V-CSC on concentrate line behind process filters, V-CSD on mixed permeate line behind filters	To isolate the composite sampler from the four sampling points as needed.

A similar system was used to control and record pH. A probe was installed in the tank and connected to a control system (PHIS-1). On rising pH the "low" contact actuated an acid pump (P-7) which transferred sulfuric acid from a drum to the surge tank. This same contact, utilizing a "dead band" of 0.1 pH unit, stopped the acid pump on falling pH. A "high" alarm, set at a pH above the normal operating range, would shut down the system and sound an audible alarm if pH in the Feed Tank rose above a preset limit.

Feed from the Feed Tank was pumped through a five-stage ultrafiltration system which contained spiral-wound membrane modules. Feed from the feed tank was pumped by a small booster pump (P-6) through a cumulative flow meter (FM-1) to the suction of the Stage 1 pump (P-1). Flow from the pump was through a throttle valve (V-1I) and a back-flushing filter system (F-1A and F-1B) for removal of residual suspended solids, prior to introduction into the ultrafiltration unit. Filter inlet and outlet pressures were measured (PI-6 and PI-1I). An automatic backwashing cycle was triggered by a differential pressure switch (PS-F) installed between the filter inlet and outlet lines. A pressure relief valve (V-11) installed on the upstream side of the filter, but controlled by a sensing element on the downstream side, maintained a constant pressure at the filter outlet. Recirculation of feed through the back pressure regulator was to the Stage 1 pump suction. After the filter, flow was through a check valve (V-12) into three parallel passes of membrane cartridges. Membrane Assemble 1-A initially contained Eastman Kodak spirals (3) and assemblies 1-B and 1-C contained TJ Engineering spirals (3 each). Permeates from each pass were collected separately and their flows were measured and sampled individually.

On the inlet side of Stage 1, pressure (PI-1I) and temperature (TI-1I) were measured, and a sample valve (V-1SI) was used for collection of Stage 1 feed. A pressure relief valve (V-1R) was provided as a safety device. The concentrate was sampled through a sample valve (V-1SE) and its flow rate (FI-1C) and pressure (PI-1E) measured. A line was provided to recirculate part of the concentrate through a "bypass valve" (V-13). Stage 1 could operate with or without recirculation through V-13.

Operation of the subsequent membrane stages was always with recirculation, since this was required to maintain a feed flow rate through the membrane cartridges at or above about 4 gpm. Flow on a "once-through" basis would fall below this level, and recirculation was required to achieve satisfactory operation.

Flow to Stages 2 through 5 was introduced into the suction side of the circulation pump for each stage. A low pressure switch (PS-2) was installed to shut down these stages should an upset in flow occur.

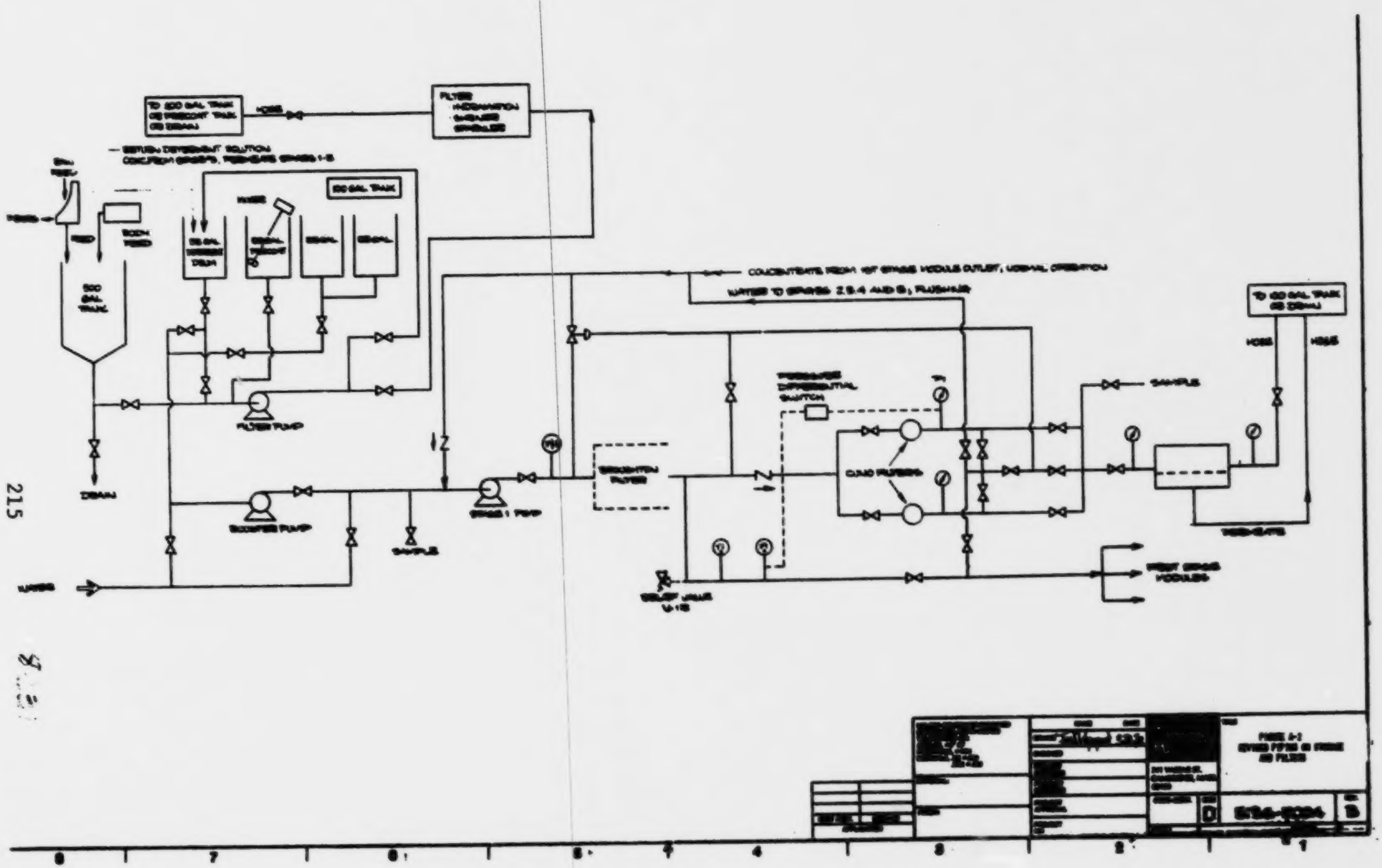
For each stage (Stages 2 through 5 are identical) flow from the booster pump passed through a throttle valve into the membrane cartridges. Pressure and temperature were measured on the inlet to the membranes and pressure and flow rate on the outlet. Pressure relief valves were provided as safety devices should overpressurization occur. Sample valves allowed collection of feed and concentrate samples for each stage. Permeate flows were individually sampled and collected, and their flow rates measured. Each stage had an internal recirculation loop which permitted maintenance of the desired flow rate through the membrane cartridges. Initially, Stages 2 and 3 contained TJ Engineering cartridges (3 each) and Stages 4 and 5 contained Gulf Environmental Systems cartridges (2 each).

The final concentrate from Stage 5 was passed through a flow control valve and flow meter (FI-C) prior to collection for incineration tests or discharge.

Each stage also had a means of introducing pressurized premeate to the feed side of the membrane cartridges for wet storage during system shutdown. The system for supplying pressurized permeate to the membrane modules was quite simple. Permeate from the five stages was manifolded and introduced into a permeate surge tank (T-2). A permeate pump (P-8) automatically pumped permeate to the membranes for each stage. Flow to the membranes was through check valves, so that when the system was operating high-pressure feed could not be pumped into the permeate system. During shutdown the permeate surge tank remained filled since all permeate fed to the membrane system was returned as permeate. During operation, permeate passed through the surge tank and overflowed to drain.

Membrane areas were approximately 300 ft^2 for Stage 1 and 100 ft^2 for each of Stages 2 through 5. Total membrane area was approximately 700 ft^2. At a nominal membrane flux of 15 gal./day/ft^2 (gfd) the plant had a capacity of 10,500 gpd.

A revised pretreatment system flow schematic is given in Figure A-2. The changes were made to permit installation of various filters, a cleaning system, and once-through flushing of the membrane shells.


TO 100 GAL TANK OR PRECOAT TANK OR DRAIN
HOSE
FILTER
100 GAL TANK
500 GAL TANK
FEED
FILTER PUMP
DRAIN
BOOSTER PUMP
WATER
SAMPLE
STAGE 1 PUMP
CONCENTRATE FROM 1ST STAGE MODULE OUTLET, NORMAL OPERATION
WATER TO STAGES 2,3,4 AND 5; FLUSHING
PRESSURE DIFFERENTIAL SWITCH
DUAL FILTERS
RELIEF VALVE V-15
FIRST STAGE MODULES
SAMPLE
TO 100 GAL TANK OR DRAIN
HOSE
HOSE
PERMEATE
D
8
7
6
5
4
3
2
1

8 ...

APPENDIX B

ULTRAFILTRATION PILOT PLANT
SPIRAL MEMBRANE CARTRIDGE IDENTIFICATION

Preceding page blank

APPENDIX B

ULTRAFILTRATION PILOT PLANT SPIRAL MEMBRANE CARTRIDGE IDENTIFICATION

UF Cartridge Location / Date		8-19-72		8-22-72		8-30-72		8-31-72	
Stage	Cartridge Position	Type	#	Type	#	Type	#	Type	#
1a	Inlet	E	1	E	1	T.J.	4	E	1
	Middle	E	2	E	2	T.J.	5	E	21
	Outlet	E	3	E	3	T.J.	6	E	3
1b	Inlet	T.J.	1	T.J.	16	T.J.	1	T.J.	1
	Middle	T.J.	2	T.J.	17	T.J.	2	T.J.	21
	Outlet	T.J.	3	T.J.	18	T.J.	3	T.J.	3
1c	Inlet	T.J.	4	T.J.	13	E		Gulf	1
	Middle	T.J.	5	T.J.	14	E			
	Outlet	T.J.	6	T.J.	15	E		Gulf	2
2	Inlet	T.J.	7	T.J.	7	T.J.	19	T.J.	19
	Middle	T.J.	8	T.J.	8	T.J.	20	T.J.	20
	Outlet	T.J.	9	T.J.	9	T.J.	21	T.J.	21
3	Inlet	T.J.	10	T.J.	10			T.J.	10
	Middle	T.J.	11	T.J.	11			T.J.	11
	Outlet	T.J.	12	T.J.	12			T.J.	12
4	Inlet	Gulf	1	Gulf	1			T.J.	13
	Middle	Gulf	2	None				T.J.	14
	Outlet			Gulf	2			T.J.	15
5	Inlet	Gulf	3	Gulf	3			Gulf	3
	Middle	Gulf	4	None					
	Outlet			Gulf	4			Gulf	4

Lot numbers of the first set of membrane cartridges were not recorded, and they are therefore numbered starting from No. 1. From 11/1/72 all cartridges are identified by manufacturer's lot number.

E = Eastman

Entries denote when cartridges were changed; if no entry is given, then the cartridge was not changed.

ULTRAFILTRATION PILOT PLANT SPIRAL MEMBRANE CARTRIDGE IDENTIFICATION

UF Cartridge Location	Date	11-1-72		11-3-72		11-5-72		11-18-72	
Stage	Cartridge Position	Type	#	Type	#	Type	#	Type	#
1a	Inlet	T.J.	4713						
	Middle	"	4723						
	Outlet	"	4724						
1b	Inlet	"	4715						
	Middle	"	4709						
	Outlet	"	4711						
1c	Inlet	"	4725			T.J.	3747		
	Middle	"	4722			T.J.	4725		
	Outlet	"	4719			T.J.	4722		
2	Inlet	"	4717						
	Middle	"	4716						
	Outlet	"	4714						
3	Inlet	"	4708						
	Middle	"	4736						
	Outlet	"	4712						
4	Inlet	"	4737					T.J.	3752
	Middle	"	4735					T.J.	4731
	Outlet	"	4731					T.J.	4735
5	Inlet	"	4718	T.J.	4718	Gulf	3		
	Middle	"	4720	T.J.	4720				
	Outlet	"	4721	T.J.	4523	Gulf	4		

E = Eastman

APPENDIX B

ULTRAFILTRATION PILOT PLANT SPIRAL MEMBRANE CARTRIDGE IDENTIFICATION

UF Cartridge Location Date		12-8-72		12-19-72		12-21-72		1-4-72	
Stage	Cartridge Position	Type	#	Type	#	Type	#	Type	#
1a	Inlet	T.J.	1948	T.J.	4715	T.J.	4715		
	Middle	"	1970	"	4709	"	4709		
	Outlet	"	3749	"	4711	"	4711		
1b	Inlet	"	4715	"	1948	"	2026		
	Middle	"	4709	"	1970	"	4283		
	Outlet	"	4711	"	3749	"	4395		
1c	Inlet	"	3747			"	3747		
	Middle	"	4725			"	4725		
	Outlet	"	4722			"	4722		
2	Inlet	"	4717			"	4717	T.J.*	4829
	Middle	"	4716			"	4716	T.J.*	4828
	Outlet	"	4714			"	4714	T.J.*	4827
3	Inlet	"	4708			"	4708		
	Middle	"	4736			"	4736		
	Outlet	"	4712			"	4712		
4	Inlet	"	3752			Gulf	1		
	Middle	"	4731						
	Outlet	"	4735			Gulf	2		
5	Inlet	Gulf	3			Gulf	3		
	Middle								
	Outlet	Gulf	4			Gulf	4		

*Wide channel corrugated spacer cartridges

E = Eastman

ULTRAFILTRATION PILOT PLANT SPIRAL MEMBRANE CARTRIDGE IDENTIFICATION

UF Cartridge Location	Date	2-8-73		2-10-73		2-14-73			
Stage	Cartridge Position	Type	#	Type	#	Type	#	Type	#
1a	Inlet	T.J.	1970			T.J.	1950		
	Middle	"	3748			T.J.	4722		
	Outlet	"	2209			T.J.	4714		
1b	Inlet	"	2026						
	Middle	"	4283						
	Outlet	"	4395						
1c	Inlet	"	3751B	T.J.	3749				
	Middle	"	3782	T.J.	3782				
	Outlet	"	3751A	T.J.	3751B				
2	Inlet	"	4829						
	Middle	"	4828						
	Outlet	"	4827						
3	Inlet	"	4708						
	Middle	"	4736						
	Outlet	"	4712						
4	Inlet	"	4731						
	Middle	"	4721						
	Outlet	"	4737						
5	Inlet	"	4735						
	Middle	"	4720						
	Outlet	"	4718						

E = Eastman

APPENDIX C

DETAILS OF FILTERS USED IN PILOT PLANT PROGRAM

Preceding page blank

- **Can save by recovering solids formerly lost in effluents**
- **Reduces operating costs and improves efficiency in process separations and in pollution control**
- **Minimal maintenance and no power costs—there are no pumps, motors, nozzles or moving parts**
- **Provides fast return on investment (often within months)**
- **Unique screen is self-cleaning, non-clogging and puncture-proof. Needs little or no attention**
- **Easy to install. Saves space**

The patented C-E Bauer Hydrasieve™ is a simple, highly efficient screening device for removing solids from low consistency slurries.

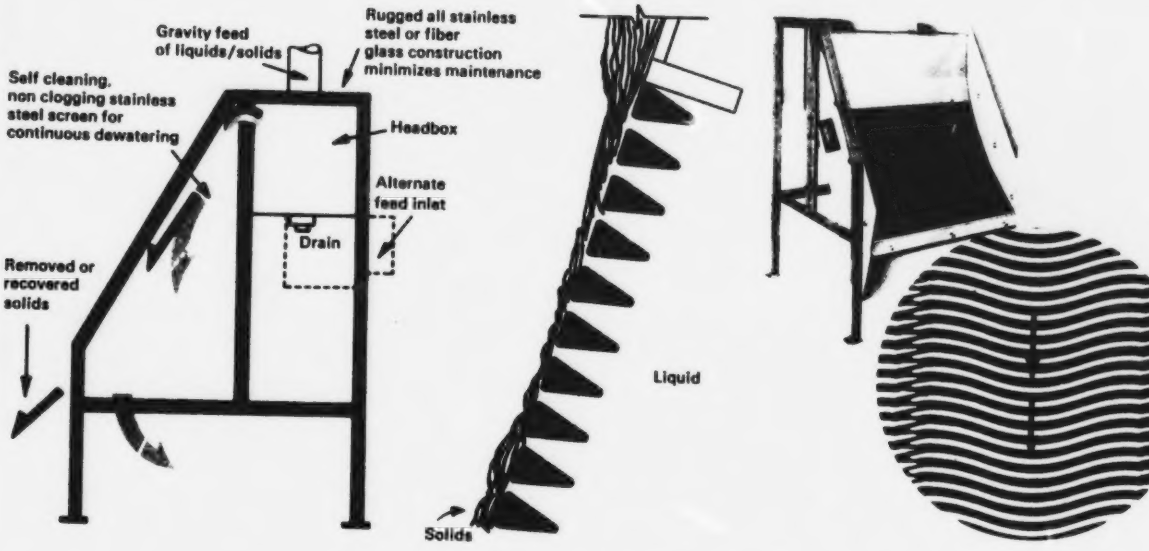
Exclusive design features and the unit's ability to operate continuously for extended periods without attention make the rugged Hydrasieve a reliable profit builder in a broad range of fluid removal applications. Hundreds of units are now in use.

Typical liquids/solids separation operations include dewatering, thickening, recovering usable solids, classifying, and fractionating.

Installations. Waste water treatment and pollution control systems. Municipal sewage plants. Chemical, plastic, and ceramic classification. Synthetic and natural fiber recovery. Salvaging rubber fines. Processing soup ingredients, fish, citrus fruits, etc. Recovering hog hair and other valuable solids for meat and hide processors, and similar operations.

C-E Bauer developed the Hydrasieve originally for the pulp and paper industry. In high density pulping systems, it pre-thickens pulp ahead of the Bauer Helipress*

HOW IT WORKS


Gravity feed of liquids/solids
Rugged all stainless steel or fiber glass construction minimizes maintenance
Self cleaning, non clogging stainless steel screen for continuous dewatering
Headbox
Alternate feed inlet
Drain
Removed or recovered solids
Liquid
Solids

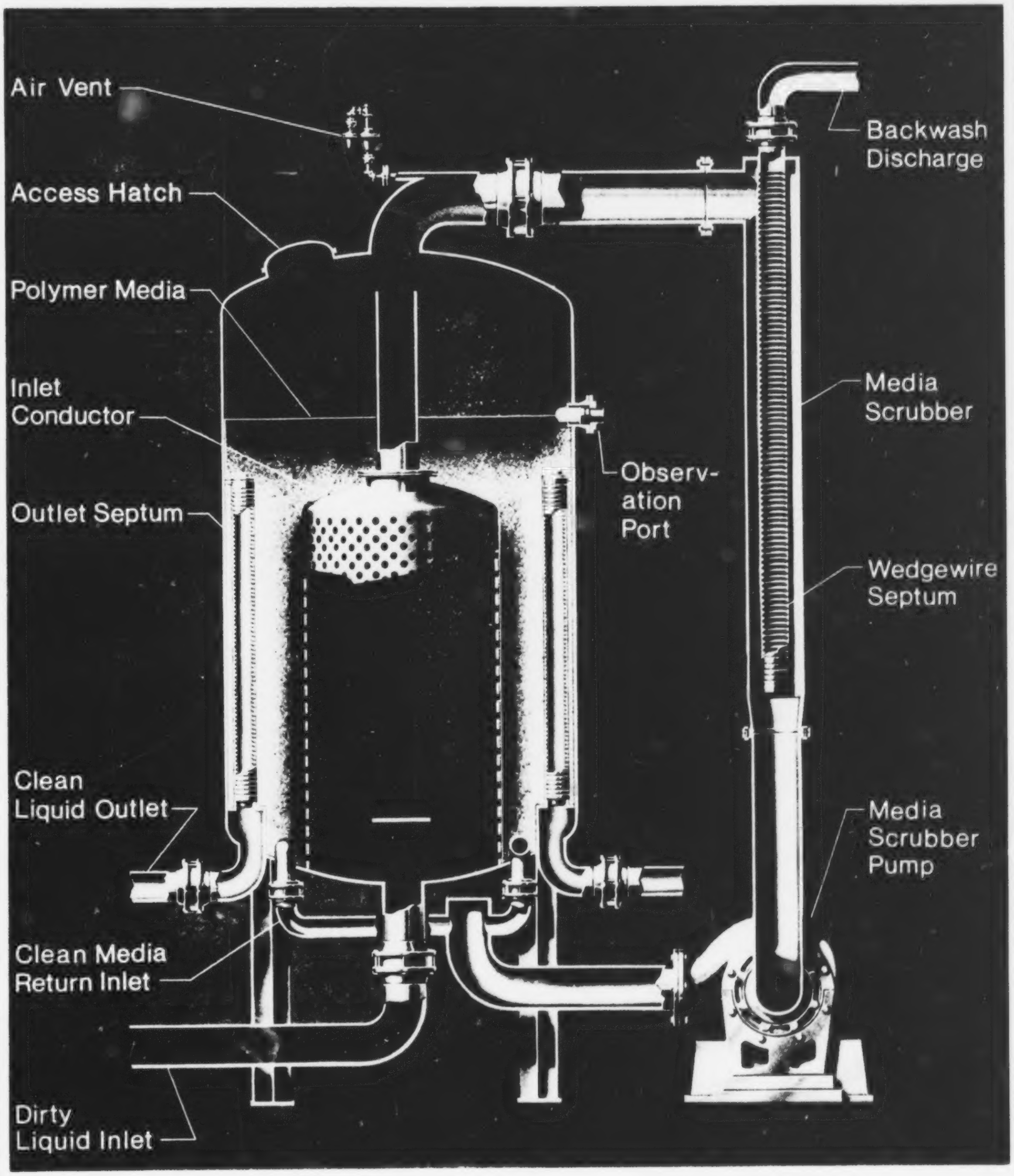
"Cleaned" effluent improves plant pollution control efficiency, lowers B.O.D., reduces sewage rates

Note how "Coanda" effect strips liquid from bottom of the stream flowing down the screen. This "wall attachment" effect accelerates fluid removal action.

Downward curve of Bauer screen bars divides the flow of slurry into separate streams between the vertical supports thus preventing clogging or blinding.

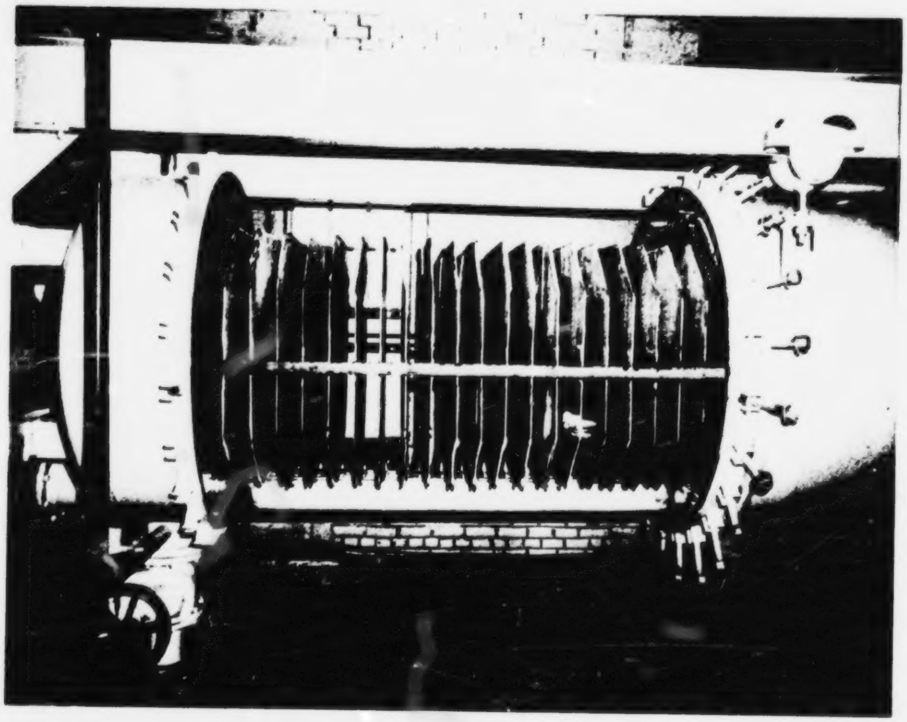
---

*The Hydrasieve is manufactured under U.S. Letters Patents No. 3,451,555 and 3,452,876, and patents pending. Canadian patent No. 835737; Great Britain 1,196,303/1,196,304; Mexico 99,076; France 1,529,448; Belgium 729,813. Other patents are pending in the United States, and foreign countries.


Air Vent
Access Hatch
Polymer Media
Inlet Conductor
Outlet Septum
Clean Liquid Outlet
Clean Media Return Inlet
Dirty Liquid Inlet
Backwash Discharge
Media Scrubber
Observ-ation Port
Wedgewire Septum
Media Scrubber Pump

the revolutionary
HYDROMATION IN-DEPTH FILTER

THE SPARKLER MCRO FILTER IS A REVOLUTIONARY DESIGN WITH THE EMPHASIS ON SIMPLICITY AND EFFICIENCY



OVERHEAD SUSPENSION DESIGN

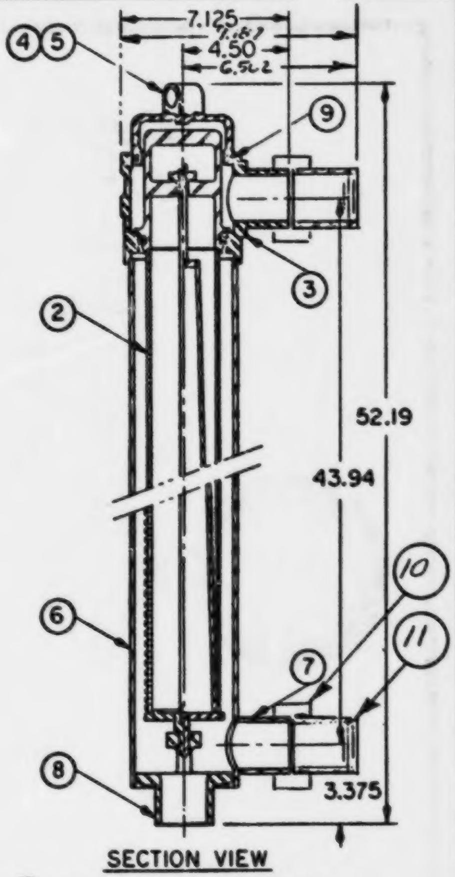
- **Engineered** for heavy duty service
- **Rugged** structural steel frame
- **Rigid** support for the tank and cover
- **Stationary** cover — unnecessary to break inlet and outlet piping to open filter
- **Retractable** tank with 4 point suspension
- **Perfect** meshing of tank and cover — positive seal
- No cake disturbance in opening — plates remain stationary
- Internal self-sluicing
- Dry cake removal either by retracting tank or internal conveyor screw
- While in operation ½ of frame space free for traffic
- Simple to completely automate
- Capacity 10 to 3000 sq. ft. filter area

F.R.

ITEM NO.	PART No. PER CONSTRUCT. MAT'L.				QUAN.	DESCRIPTION
	DWG. NO.	BRONZE	STN.STL	STEEL		
1	3002-000-	-001	-002	-003	1	FILTER ASSEMBLY
2	3001-001-	-001	-001	-001	1	BASKET ASSEMBLY
3	3000-011-	-004	-005	-006	1	TOP HOUSING
4	3001-014-	-004	-004	-004	1	SAFETY LOCK
5	-015-	-001	-002	-003	1	TOP CAP
6	-016-	001	002	003	1	CASING
7	-017-	-004	-005	-006	1	INLET CONNECTION
8	-018-	-004	-005	-006	1	BOTTOM
9	3001-019-	SEE TABULATION			1	O-RING
10	2508	C	C	C	2	CLAMP
11	675	—	A	B	2	HALF NIPPLE

O-RING MATERIAL AVAILABLE

NITRILE BUNA N TEFLON COATED	VITON TEFLON COAT	TEFLON	TEFLON ENCAPSULATED
3001-019-001	3001-019-002	3001-019-003	3001-019-004



SECTION VIEW

① FILTER ASSEMBLY

MAX. OPERATING PRESSURE ________ PSI

MAX. OPR. TEMPERATURE ________ °F

CUSTOMER ________

ADDRESS ________

CUST. ORDER No. ________

BROUGHTON ORD. No. ________

BASKET FILTER MEDIA ________

DATE SHIPPED ________

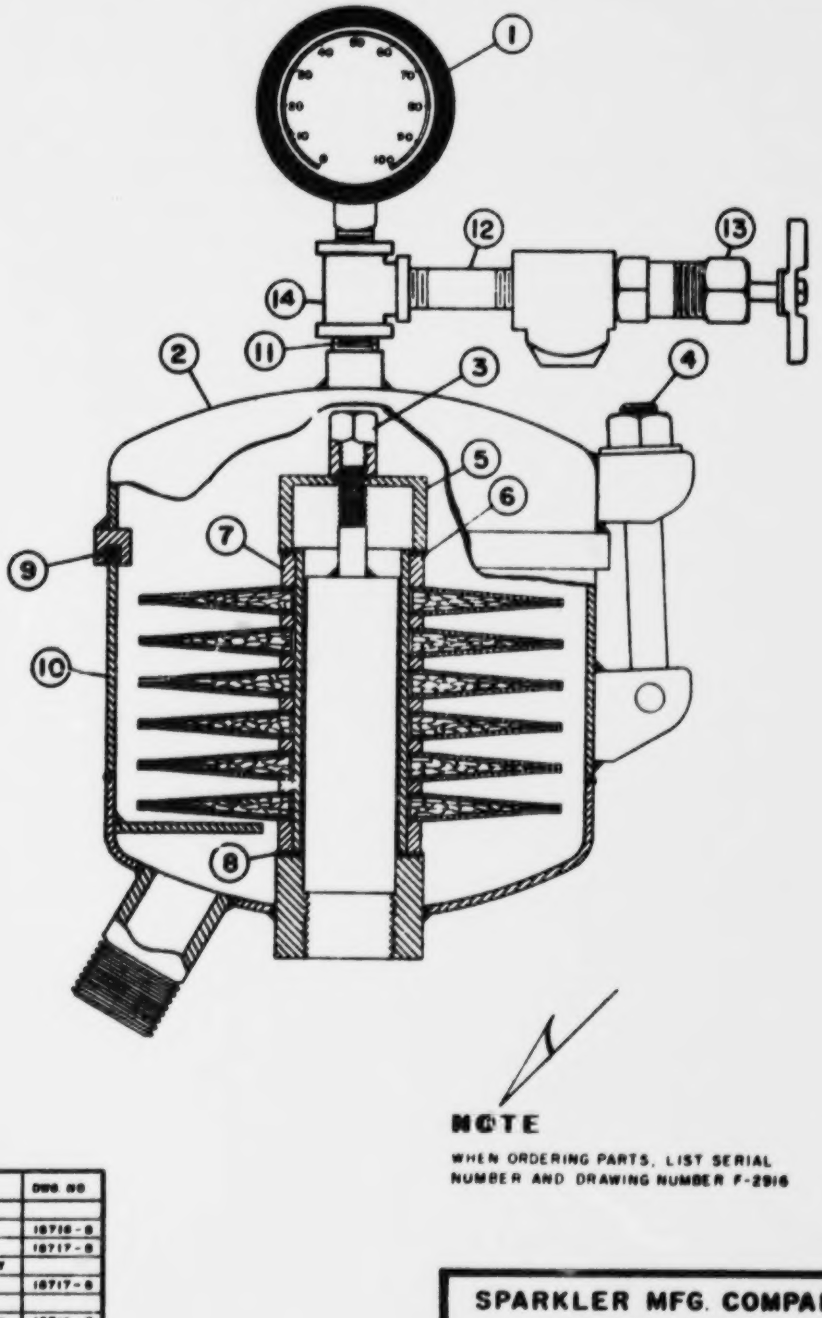
BART WILSON
H. A. Wilson Company
473 WASHINGTON STREET
WELLESLEY, MASS. 02181
(617) 237-1755

BROUGHTON CORPORATION
GLENS FALLS NEW YORK, 12801

3000 FILTER ASM-2" PLAIN END

DWG. BY: B.W.F.
CK'D. BY: RE
SCALE:
DATE: 1-13-72
DWG. NO. 3002-000-00

C-5128 F.R.



NOTE

WHEN ORDERING PARTS, LIST SERIAL NUMBER AND DRAWING NUMBER F-2916

ITEM NO.	ITEM	DWG. NO
1	PRESSURE GAUGE	
2	COVER ASSEMBLY	16716-B
3	COMPRESSION NUT	16717-B
4	SWING BOLT ASSEMBLY	
5	COMPRESSION CAP	16717-B
6	COMP CAP GASKET	
7	CARTRIDGE ASSEMBLY	16715-B
8	OUTLET GASKET	
9	COVER GASKET	
10	TANK ASSEMBLY	16716-C
11	NIPPLE	
12	NIPPLE	
13	VALVE	
14	TEE	
15		
16		

SPARKLER MFG. COMPANY
CONROE TEXAS

STANDARD L-6 PARTS LIST
"GUARDIAN" TRAP FILTER

DATE	11 5 '64	DRN BY	WEJ	F-2916
SCALE	—	CKD BY	BED	

TYPE IB

175 P.S.I. @ 250°F
¾" NPT Connections

1B1

1B2

Three-piece housing; centerpost construction. Available in one and two-high cartridge models.

STANDARD — cast iron and steel housing, asbestos shell gaskets, fiber cap nut gasket, drain plug, steel internals, or, 304 stainless steel housing and 304 stainless steel internals. Mounting pads on all models are drilled and tapped for mounting bracket.

DIMENSIONS—WEIGHTS

1B1 [illegible]	¾" ¾"	12⅞" 22⅝"	4⅛" 4⅛"	9 lbs. 12 lbs.

HOUSING CATALOG NUMBERS

1B1 B2	¾" ¾"	40292-11 40292-12	40294-11 40294-12

Mounting bracket . . . . **#35581-01**

Filter utilizes:
Micro-Klean Series G78 cartridge(s)
all Micro-Wynd cartridge(s)
Micro-Screen Series 52243 cartridge
Micro-Screen Series 52043 cartridge
Poro-Klean Series 50387 cartridge

TYPE CT

300 P.S.I. @ 200°F
¾" or 1" NPT Connections

CT-101

CT-102

Three-piece housing; ring nut construction holds sump to head. Available in one and two-high cartridge models.

STANDARD — cast brass head and ring nut, drawn 304 stainless steel sump, Buna N head gasket, 302 stainless internals. Heads with ¾" connections have mounting pads drilled and tapped for mounting bracket. Also available with a 304 stainless steel cast head (ring nut nickel plated.)

DIMENSIONS—WEIGHTS

CT-101 CT-102	¾" 1"	12$\frac{5}{16}$" 22⅝"	2$\frac{15}{16}$" 3$\frac{5}{16}$"	6 lbs. 7½ lbs.

HOUSING CATALOG NUMBERS

CT-101 CT-101	¾" 1"	40245-01 40469-02	40245-06 —
CT-102 CT-102	¾" 1"	40245-03 40469-01	40245-07 —

Mounting bracket . . . . **#35581-03**

Filter utilizes:
Micro-Klean Series G78 cartridge(s)
all Micro-Wynd cartridge(s)

ORDER CARTRIDGES SEPARATELY

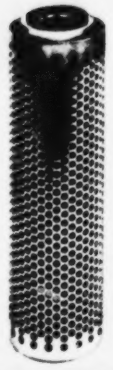
THE RIGHT FILTER FOR ANY FLUID

Liquid and gas filters of AMF Cuno design are available from your distributor in several types. Each filter type is described below with detailed information further in the catalog.

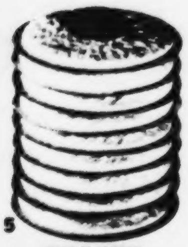
1
MICRO-KLEAN II

2
MICRO-WYND II

4
PORO-KLEAN


3
MICRO-SCREEN

6
AUTO-KLEAN


5
CUNO-PORE

1 **MICRO-KLEAN II** . . . depth type, disposable fiber cartridge in micronic ratings. Ideal for removing contaminant which is fibrous, abrasive or gelatinous in character. Typical applications include gas, alcohols, glycols, coolants, fuels, oils, lubricants, cosmetics, paints and varnishes, syrups, compressed air, water. See pages 4 and 5. For Micro-Klean air line filters see pages 14 and 15.

2 **MICRO-WYND II** . . . depth type, disposable wound cartridge in ratings from 1 to 350 microns. Intended for use where Micro-Klean fibers or resin binders are not compatible with the fluid, or, where a wound type cartridge is preferred. Typical applications include plating solutions, organic solvents, waxes, detergents, plastics, resins, deodorants, animal and vegetable fats and oils, metal cleaning solutions. See pages 6 and 7.

3 **MICRO-SCREEN** . . . surface type, metal screen cartridge —cleanable and reusable—in micronic and screen mesh ratings. Ideal filter media for final protection in ultra-clean fluid systems . . . offering complete freedom from media migration. Also intended for applications involving high temperature and corrosive conditions. Typical applications include steam, strong acids, concentrated alkalis, strong reducing agents and other chemicals which react with fiber type cartridges. See pages 8 and 9.

4 **PORO-KLEAN** . . . depth type, sintered metal cartridge— cleanable and reusable in 5, 10, 20 and 40 micron ratings. Used in place of Micro-Screen when contaminant is gelatinous or fibrous in character. Also recommended for heavy viscous fluids. Typical applications include steam, air, water, solvents, polymers, acids, cellulose solutions, process gases. See pages 8 and 9.

5 **CUNO-PORE** . . . depth type, disposable media in cartridge and disc form. Will remove contaminant which is fibrous, abrasive or gelatinous in character. Possessing natural dessicating properties, it is ideal for removing trace quantities of water from oil. Recommended for free-flowing liquids where optical clarity is of primary importance. Cartridge type filters are used on dielectric insulating oils, coolants, detergents, cosmetics, light lubricants, fuels, degreasers and chemicals.
See pages 16 - 19.

6 **AUTO-KLEAN** . . . edge type, all metal filter. One turn of the handle cleans cartridge and restores full flow. Ideal for removing particles as small as 36 microns (.0015"). Typical applications include paint, adhesives, resins, greases, inks, tar, cellulose solutions, waxes, soaps, hydrocarbon oils, fuels and lube oils. See pages 20-23

APPENDIX D

MECHANICAL PROBLEMS OF DIFFERENT SPIRAL MEMBRANE CARTRIDGES

APPENDIX D

MECHANICAL PROBLEMS OF DIFFERENT SPIRAL MEMBRANE CARTRIDGES

Date	Operating Information			Membrane Cartridge Characteristics		Problems	Comments
	Stage	Cartridge Number	Cartridge Position	(ΔP), psi	Rejection		
11-5-72	1a	T.J. 4713 4723 4724	Inlet Middle Outlet	50-60	O.K.	Brine seals loose fit	Reinforced brine seal
12-7-72	1a	T.J. 4713	Inlet			Brine seal flipped	May have happened around 12-2 when ΔP was high
2-7-73	1a	T.J. 4715 4709 4711	Inlet Middle Outlet		O.K.	Brine seals flipped	May have occurred around 1-4-73 when ΔP suddenly dropped from a high value to a low one
2-13-73	1a	T.J. 1970 3748 2209	Inlet Middle Outlet		Low	O-ring leak	All three cartridges were tested individually and found to give good color rejection
8-21-72	1b	T.J.			Low	Cartridge failure*	Membrane replaced on 8-22-72
11-15-72	1b	T.J. 4715	Inlet	50-60	O.K.	Brine seals weak	Reinforced brine seal with additional tapes
12-7-72	1b	T.J. 4715	Inlet	50-60	O.K.	Brine seal flipped	

APPENDIX D
(continued)

MECHANICAL PROBLEMS OF DIFFERENT SPIRAL MEMBRANE CARTRIDGES

Date	Operating Information			Membrane Cartridge Characteristics		Problems	Comments
	Stage	Cartridge Number	Cartridge Position	(ΔP), psi	Rejec-tion		
12-20-72	1b	T.J. 1948	Inlet		Low	Cartridge failure*	Membrane replaced on 12-21-72
8-21-72	1c	T.J.			Low	Cartridge failure*	Membrane replaced on 8-21-72
9-13-72	1c	T.J.			O.K.	Brine seal weak	Reinforced brine seal with tapes
11-15-72	1c	T.J. 4722	Inlet		Low	O-ring failure	Replaced O-ring and rejection became normal
1-4-73	2	T.J. 4717 4714	Inlet Outlet		O.K.	Brine seals squeezed out, flow leaking underneath brine seals	May have happened between 12-3-72 and 12-5-72 when the (ΔP) was high
12-6-72	3	T.J. 4708	Inlet		O.K.	Brine seal flipped; other two cartridges were O.K.	

APPENDIX D
(continued)

MECHANICAL PROBLEMS OF DIFFERENT SPIRAL MEMBRANE CARTRIDGES

Date	Operating Information			Membrane Cartridge Characteristics		Problems	Comments
	Stage	Cartridge Number	Cartridge Position	(ΔP), psi	Rejection		
10-26-72	4	T.J.			Low	Unknown reasons	
10-31-72	4	T.J.			Low	Brine seals weak	
11-16-72	4	T.J. 4737 4735 4731	Inlet Middle Outlet		Low	O-ring failure	All three cartridges were O.K. when tested individually later on. Replaced cartridges.
12-19-72	4	T.J. 3746	Inlet			Brine seal flipped	(ΔP) rose to 50 psi before it happened
12-21-72	4	T.J. 4724	Middle			O-ring failure	Replaced all cartridges by two Gulf cartridges
2-7-73	4	Gulf	Inlet		O.K.	Brine seal weak	Brine seal of the second Gulf cartridge was O.K.
11-1-72	5	T.J. 4721	Outlet		Low	O-ring leak	Slot too wide. Replaced cartridge.

APPENDIX D
(continued)

MECHANICAL PROBLEMS OF DIFFERENT SPIRAL MEMBRANE CARTRIDGES

Date	Operating Information			Membrane Cartridge Characteristics		Problems	Comments
	Stage	Cartridge Number	Cartridge Position	(ΔP), psi	Rejection		
11-3-72	5	T.J. 4718	Inlet		Low	Cartridge failure*	Cartridges 4718 and 4720 were found to be O.K. during individual cartridge screening tests. Replaced all three T.J. cartridges by Gulf cartridges
		4720	Middle				
		4523	Outlet				
2-7-73	5	Gulf	Inlet		O.K.	Brine seals had loose tapes	
		Gulf	Outlet				

*Any one of the following reasons:

1. Leakage at the glue seam;
2. Leakage at the material fold adjacent to the permeate collection tube;
3. Membrane and/or backing material wrinkles causing direct leakage;
4. Pinholes in membrane.

APPENDIX E

SLIME ANALYSIS

A sample of the material flushed from a fouled membrane cartridge was analyzed. The material was de-cationized using Amberlite IR-120 resin. The flow sheet showing the make-up of the sample and the fractions obtained is as follows:

The six fractions from the cation column were examined. The results leave some strange unanswered questions.

Fractions "A" and "C" should be the same. When pyrolyzed, the chromatograms closely matched. However, the TGA showed degradations at 270° and 520° with a 10% white ash for "A" and degradations at 270° and 440° with 1.3% white ash for "C". The difference in ash content is unexplainable; especially since neither sample should contain any inorganic salts.

The chromatograms from the pyrolysis of these fractions closely match the pyrolysis chromatogram for glucose. The pyrolysis by-products can be grouped into three groups. This is a 600° pyrolysis.

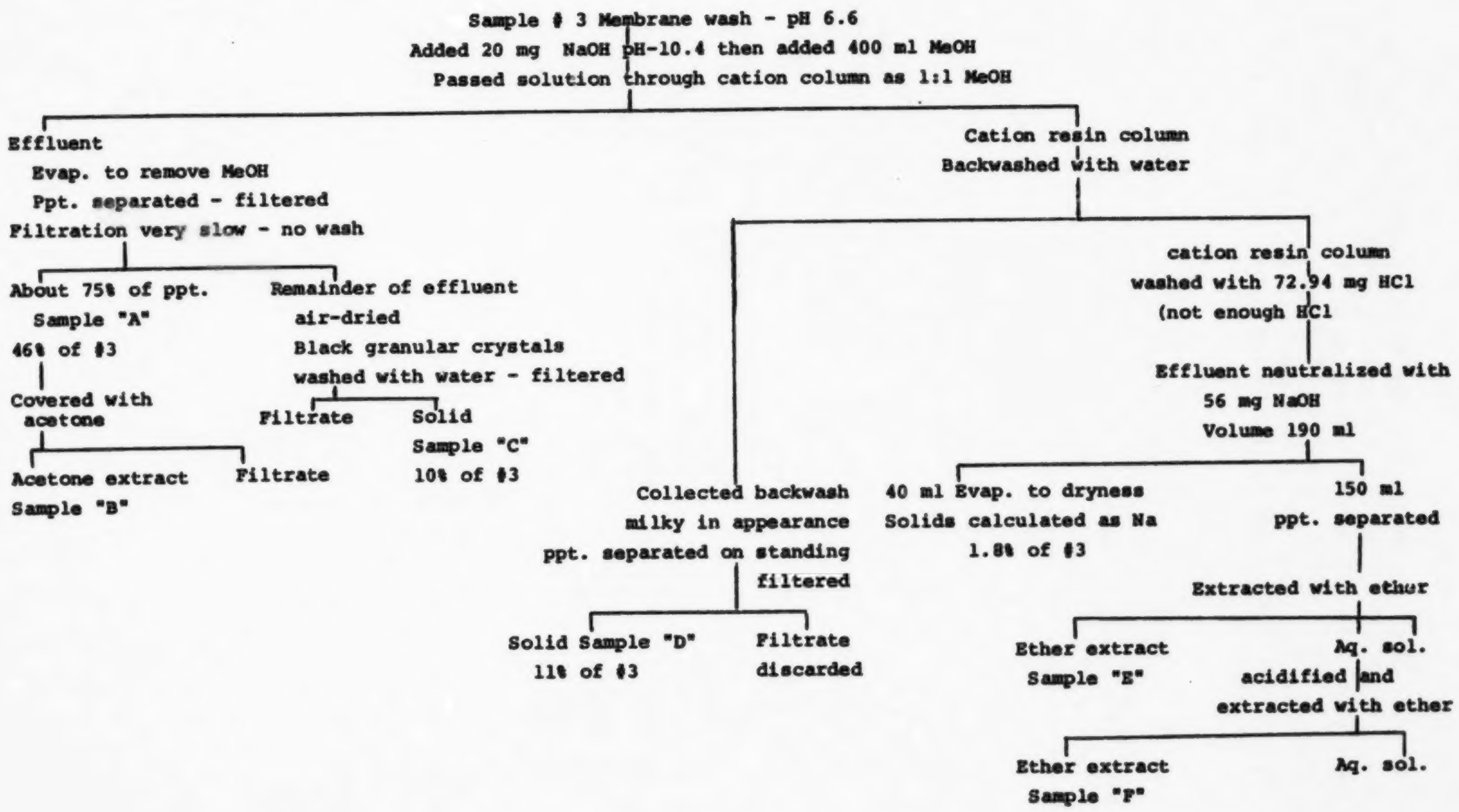
1. Very volatile - retention times 0.6 to 2.2 min.
2. Intermediate - rentention times 6.0 to 12.3 min.
3. High boiling - retention times 14.0 to 30.0 min.

Groups 1 and 2 closely match the pyrolysis of glucose; however group 2 is definintely stronger than glucose.

When pyrolized at 950°, the chromatograms match glucose even closer. Apparently some of this material is polysaccharides in nature. It should be noted that even though a similarity exists to a pyrolysis of cellulose, several peaks are different.

Fraction D. The 600° pyrolysis of "D" match as far as groups 1 and 2 are concerned; however the high boiling group of compounds are completely different. A sample of the crystals from the concentrate was dissolved in water and acidified. The precipitate was filtered. The pyrolysis of this solid matches exactly fraction "D". TGA showed degradation at 300, 500, and 650°. Ash was 25% white.

Preceding page blank


Sample # 3 Membrane wash - pH 6.6
Added 20 mg NaOH pH-10.4 then added 400 ml MeOH
Passed solution through cation column as 1:1 MeOH
Effluent
Evap. to remove MeOH
Ppt. separated - filtered
Filtration very slow - no wash
About 75% of ppt.
Sample "A"
46% of #3
Covered with acetone
Acetone extract
Sample "B"
Filtrate
Remainder of effluent
air-dried
Black granular crystals
washed with water - filtered
Filtrate
Solid
Sample "C"
10% of #3
Cation resin column
Backwashed with water
Collected backwash
milky in appearance
ppt. separated on standing
filtered
Solid Sample "D"
11% of #3
Filtrate
discarded
cation resin column
washed with 72.94 mg HCl
(not enough HCl
Effluent neutralized with
56 mg NaOH
Volume 190 ml
40 ml Evap. to dryness
Solids calculated as Na
1.8% of #3
150 ml
ppt. separated
Extracted with ether
Ether extract
Sample "E"
Aq. sol.
acidified and
extracted with ether
Ether extract
Sample "F"
Aq. sol.

Fraction B. The chromatogram of this fraction was very poor. A group of 12 to 15 materials with an estimated boiling range 230 to 300°. When reacted to form the trimethylsilyl derivative, some differences are noted. The IR showed strong aliphatic and carbonyl. The extract also contained some hydroxyl absorption; but spectra quality is poor.

Fraction E. IR showed strong aliphatic, especially CH_2. Also minor indications of hydroxyl (extremely weak), carbonyl (one only), and aromatic (possibility of a little phenylphthalein - used as an indicator). The GC showed several distinct peaks and a group of lesser peaks. Slight changes occur when the fraction is derivatized.

Fraction F. This fraction chromatographs similar to fraction "B", except that a very large distinct peak is found which matches one of the peaks in "E". This material does not appear to form any derivative. These materials would all be very high boiling. The IR showed strong aliphatic (CH_2 and CH_3). Also minor indications of hydroxyl, carbonyl (2 bands) and aromatic. "E" appeared to be more contaminated than "F", although the single carbonyl indicates that at least one component is absent in "E". Other than the carbonyl difference, both "E" and "F" present very similar spectra. The strong aliphatic indicates a hydrocarbon possibility such as kerosene. Disregarding the fact that such a material should not be in "F", the GC would indicate that if hydrocarbons were present, it would have to be C-15 to 18.